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MECHANICAL BEHAVIOR OF 18Ni (300)
MARAGING STEEL DURING RAPID HEATING
AND AT ELEVATED TEMPERATURES

C. W. Austin, Jr.

Army Missile Command
Redstone Arsenal, Alabama

12 December 1973

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(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1. ORIGINATING ACTIVITY (Corporate author) US Army Missile Research, Development and Engineering Laboratory US Army Missile Command Redstone Arsenal, Alabama 35809		2a. REPORT SECURITY CLASSIFICATION Unclassified	
3. REPORT TITLE MECHANICAL BEHAVIOR OF 18Ni (300) MARAGING STEEL DURING RAPID HEATING AND AT ELEVATED TEMPERATURES		2b. GROUP NA	
4. DESCRIPTIVE NOTES (Type of report and inclusive dates) Technical Report			
5. AUTHOR(S) (First name, middle initial, last name) C. W. Austin, Jr.			
6. REPORT DATE 12 December 1973		7a. TOTAL NO. OF PAGES 44	7b. NO. OF REFS 21
8a. CONTRACT OR GRANT NO. b. PROJECT NO. (DA) 1T061102B32A AMC Management Structure Code No. c. 611102.11.85500 d.		8b. ORIGINATOR'S REPORT NUMBER(S) RL-73-10 8d. OTHER REPORT NO(S) (Any other numbers that may be assigned this report)	
10. DISTRIBUTION STATEMENT Approved for public release; distribution unlimited.			
11. SUPPLEMENTARY NOTES None		12. SPONSORING MILITARY ACTIVITY Same as No. 1	
13. ABSTRACT The mechanical behavior of 18Ni (300) maraging steel was determined in constant loading rate, short-time elevated temperature tensile tests at loading rates of 4000 to 400,000 psi/sec at temperatures from 75° to 1200°F and in heating rate tests with concurrent constant loads equivalent to 25 to 85 percent of the room temperature yield strength for heating rates of 50° to 400°F/sec. A linear relation between strength and the logarithm of heating rate and loading rate existed for all test parameters. Inverse loading rate dependence and low heating rate dependence of strength in the 600° to 1000°F temperature range is attributed to dynamic strain aging. Yield strengths obtained at temperatures above 600°F in the heating rate tests were significantly lower than those obtained in tensile tests at these temperatures. Aging this steel at either 850°F or 900°F for 3 hours did not have a significant overall effect on the mechanical behavior, but the higher temperature age did result in a slight improvement in strength in tensile tests in the 600° to 900°F temperature range and slightly better resistance to creep in the 900° to 1100°F temperature range in heating rate tests.			

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14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
18Ni (300) maraging steel Mechanical behavior Elevated temperatures Rapid heating Heating rate dependence Loading rate dependence Strain rate sensitivity Dynamic strain aging Aging kinetics						

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1. Introduction

Previous experimental investigations [1-3] have indicated that the strength and load-carrying ability of 18Ni (250) and 18Ni (300) maraging steels are significantly impaired when these steels are subjected to rapid heating under concurrent constant tensile loading. Under these conditions yielding and failure occur at lower temperatures than predicted from yield and ultimate strengths determined in conventional elevated temperature tensile tests. It has been proposed that these steels exhibit a negative heating rate dependence when stressed to at least 70 percent of their room temperature yield strengths and heated to failure under these loading conditions [3]. The heating rate dependence is considered to be negative because for a particular stress, yielding and failure occur at lower temperatures as the heating rate is increased. The negative heating rate dependence of these steels may be analogous to the negative strain rate dependence observed by Kendall [4] for 18Ni (300) maraging steel at 600°F. He attributed the decrease in yield strength with increasing strain rate at this temperature to stress aging.

A negative heating rate dependence could also result from thermodynamic instability of the martensite matrix or intermetallic precipitates during the rapid heating test procedure. While no evidence of mechanical instability of these steels exists for temperatures up to the aging temperature for material aged at 900°F, the detrimental effect of aging at temperatures below 900°F has been reflected in all grades of 18Ni maraging steel through reduced delayed fracture resistance, response to variations in strain rate, and increased susceptibility to subcritical crack growth [5-7].

The detrimental effects of aging these steels at temperatures below 900°F have generally been ascribed to a critical change in the precipitation hardening reaction at these temperatures. Peters [8] has found that aging these steels at temperatures below 900°F results in the formation of an unstable matrix precipitate along with stable dislocation-nucleated precipitates. On heating to 900°F, the unstable precipitate dissolves in solid solution and is subsequently precipitated in the form of the stable precipitate. This reversion process is known to cause a rapid momentary decrease in hardness, which is recovered as isothermal nucleation of the stable precipitate proceeds.

It is apparent that the loading-heating sequence used in the heating rate test procedure could significantly affect the mechanical behavior of these steels. The additional strain energy supplied by the applied stress could influence the stability of the martensite matrix and the intermetallic precipitates during heating at temperatures below the prior aging temperature. This could result in significant deviation from the mechanical behavior of these steels observed in conventional elevated temperature mechanical tests.

This investigation was undertaken to provide a comprehensive analysis of the elevated temperature mechanical behavior of 18Ni (300) maraging steel. The specific objectives of the investigation were:

- a) To establish the heating rate and loading rate dependence and sensitivity of this steel at temperatures up to 1200°F and to determine the influence of maraging parameters on this behavior
- b) To determine the influence of mechanical test procedure and maraging parameters on the elevated temperature strength and load-carrying ability of this steel
- c) To provide more realistic mechanical property data for material selection and design considerations for solid propellant missile motor cases and other structures subjected to rapid heating with concurrent loading or rapid loading at elevated temperatures.

2. Experimental

a. Material

Commercial grade, consumable-electrode vacuum remelt, mill-annealed 18Ni (300) maraging steel sheet with a nominal thickness of 0.082 inch was used in this investigation. The certified chemical composition in weight percent was 18.62 Ni, 9.00 Co, 4.75 Mo, 0.65 Ti, 0.15 Al, 0.014 Zr, 0.05 Ca, 0.006 S, 0.003 P, 0.002 B. Aging this steel at 900°F for 3 hours resulted in a 298,500 psi yield strength, 304,000 psi ultimate strength, and 4.0 percent elongation in 2 inches. Metallographic and X-ray diffraction analysis indicated that the microstructure of the material was uniform with no evidence of significant microsegregation or preferred orientation.

(1) Maraging Kinetics. The maraging kinetics of the steel were investigated by measurement of hardness changes occurring during isothermal aging in the 700° to 1000°F temperature range. The purpose of this study was to determine the character of the precipitation sequence and establish the lower time-temperature limits to achieve a microstructure that would remain stable during heating rate tests and short-time elevated temperature tensile tests at temperatures up to 1000°F.

Hardness changes corresponding to isothermal aging times at temperatures from 700° to 1000°F are shown in Figure 1. Each point on these curves represents the average hardness change determined from five Rockwell C hardness readings on three separate 1/2-inch square samples before and after isothermal aging. The scatter in the readings was ± 1.0 Rockwell C for the mill annealed sheet and ± 0.5 Rockwell C after the isothermal aging.

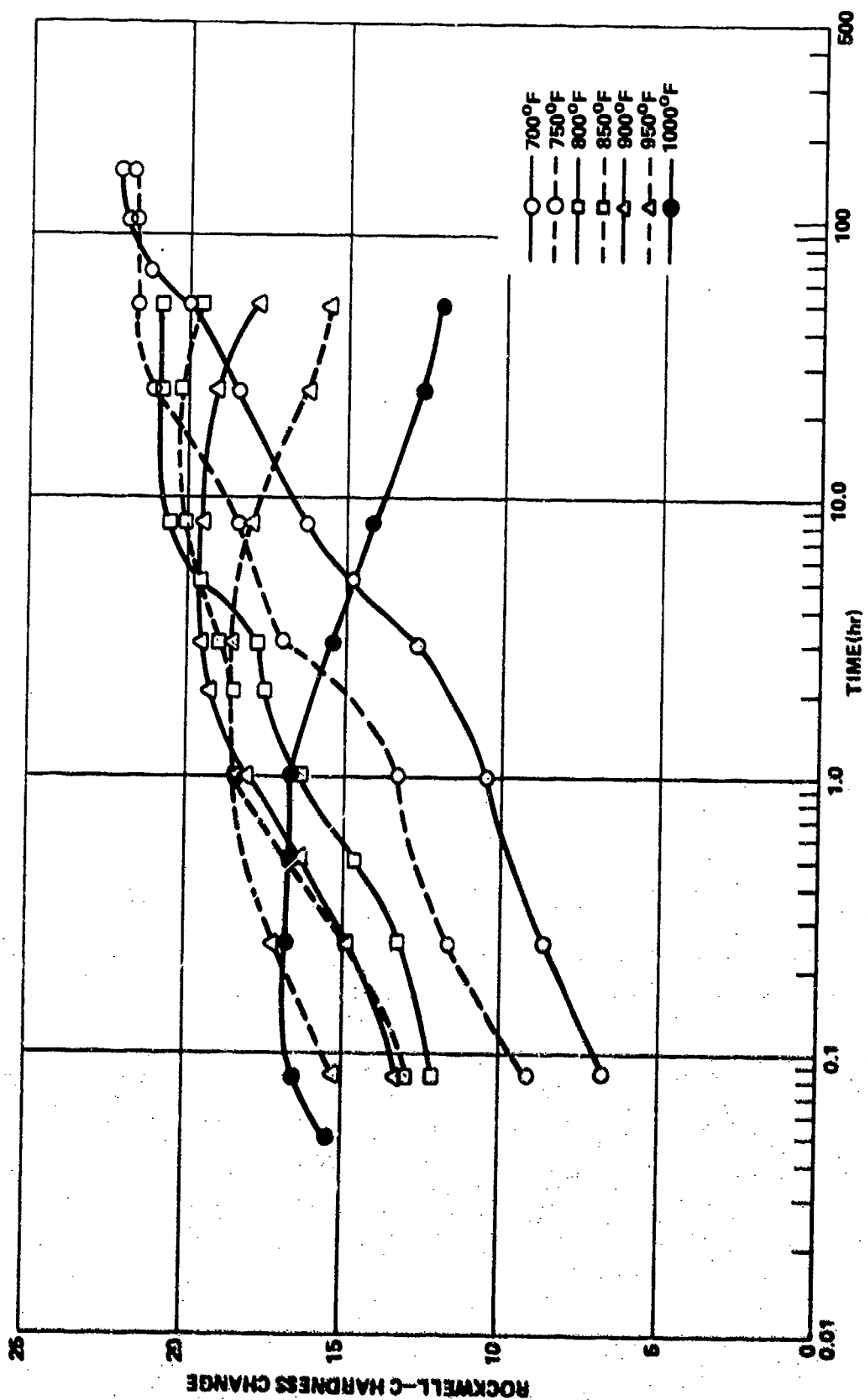


Figure 1. Influence of aging time on hardness changes during isothermal aging of 18Ni (300) maraging steel.

Two major hardening reactions are evident from the plateaus in the hardness change-time curves for the 700° to 850°F temperature range. At higher temperatures only one hardening reaction is evident. The initial minor hardening reaction evident for aging times between 0.1 and 1.0 hour at 700°F and 750°F has been attributed to carbide precipitation [9, 10]. According to Peters and Cupp [10], the initial major hardening reaction results from increased molybdenum supersaturation caused by the presence of cobalt in the steel. The higher supersaturation at lower temperatures provides the necessary driving force for simultaneous nucleation of an unstable molybdenum intermetallic matrix precipitate along with the stable dislocation-nucleated Mo_3Ni precipitate. Although other precipitates are involved in the maraging reactions, their contributions to the significant hardening reactions are apparently minor and, therefore, will not be considered here. Extended aging in the 700° to 850°F temperature range results in thermodynamic instability of the matrix-nucleated precipitate. The nuclei dissolve and are subsequently reprecipitated in the form of the stable dislocation-nucleated precipitate.

Arrhenius plots of the time to reach hardness maxima for the two hardening reactions are shown in Figure 2. Activation energies associated with these reactions were determined from the slopes of these plots. The activation energy for the initial hardening reaction was 26.5 kcal/mol. The activation energy for the second hardening reaction was 41 kcal/mol for the 700° to 800°F temperature range and 46 kcal/mol for the 850° to 1000°F temperature range. The higher rate of the second hardening reaction at temperatures below 850°F and the transition to a lower reaction rate at approximately 850°F as well as the disappearance of the first hardening reaction at approximately 875°F are evident from Figure 2. The activation energies associated with the second hardening reaction compare favorably with values of 45.3 kcal/mol obtained in internal friction measurements on an 18Ni-6Co-5Mo-0.2Ti alloy [11] and 42.8 kcal/mol obtained from electrical resistivity measurements on a commercial 18Ni maraging steel [10].

(2) Precipitate Stability and Reversion. The short-time elevated temperature stability of the hardening precipitates was determined in isothermal exposures of from 1 second to 1 hour at temperatures 50°F and 100°F above the initial aging temperature. Short isothermal exposures of 1 to 300 seconds after heating to the exposure temperature within 10 seconds were accomplished in a quartz lamp radiant heating furnace. Longer exposures were made in a heat treating furnace. Hardness changes corresponding to isothermal exposures at temperatures of 50°F and 100°F above the initial aging temperature for aging conditions considered for this study are shown in Figures 3 through 6. Each data point in these figures represents the average hardness change determined from five Rockwell C hardness readings on two separate samples.

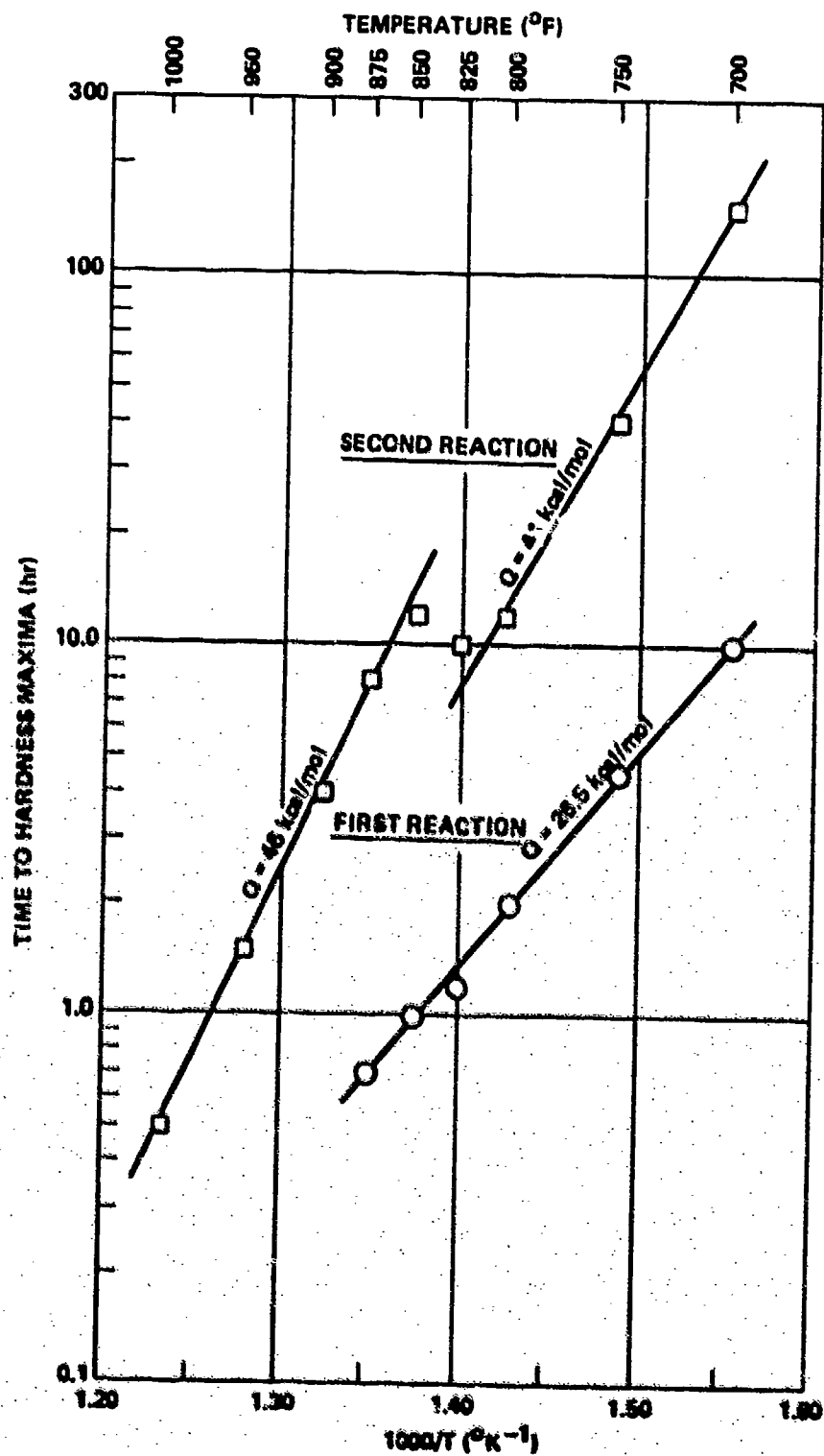


Figure 2. Arrhenius plot constructed from hardness change data for 18Ni (300) maraging steel.

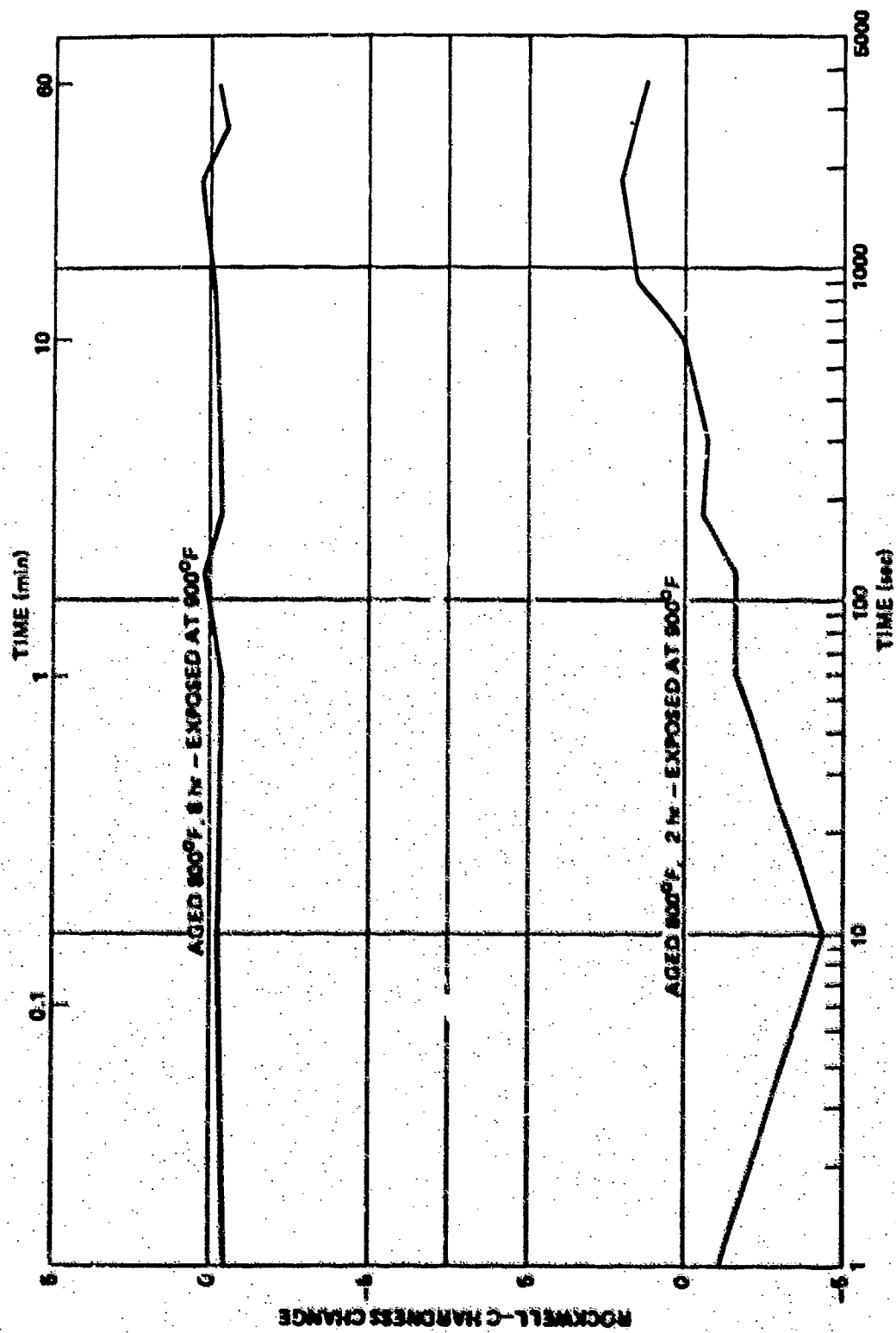


Figure 3. Hardness changes resulting from exposure of 18Ni (300) maraging steel at 900°F after aging at 800°F.

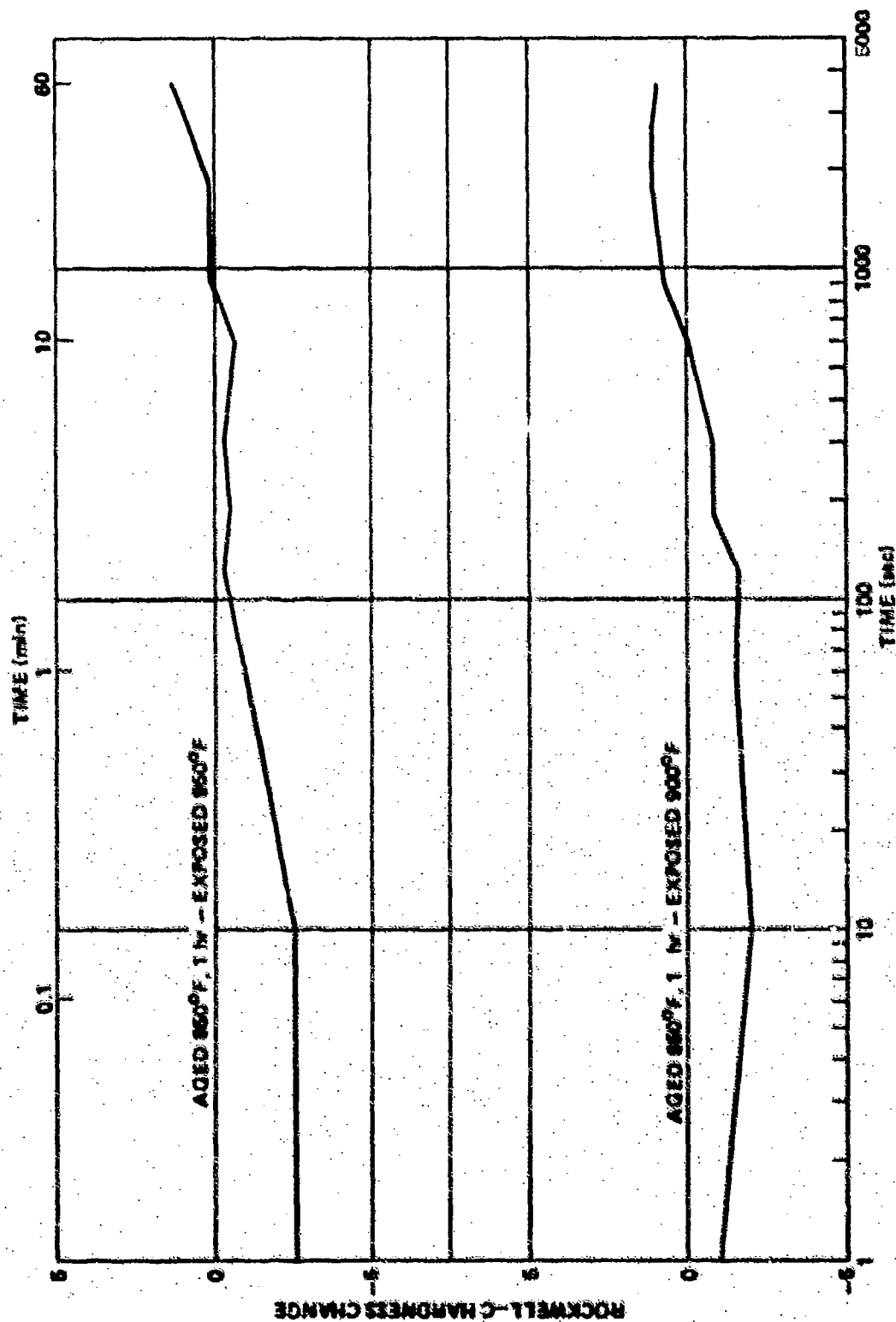


Figure 4. Hardness changes resulting from exposure of 18Ni (300) maraging steel at 950°F and 850°F after aging at 850°F for 1 hour.

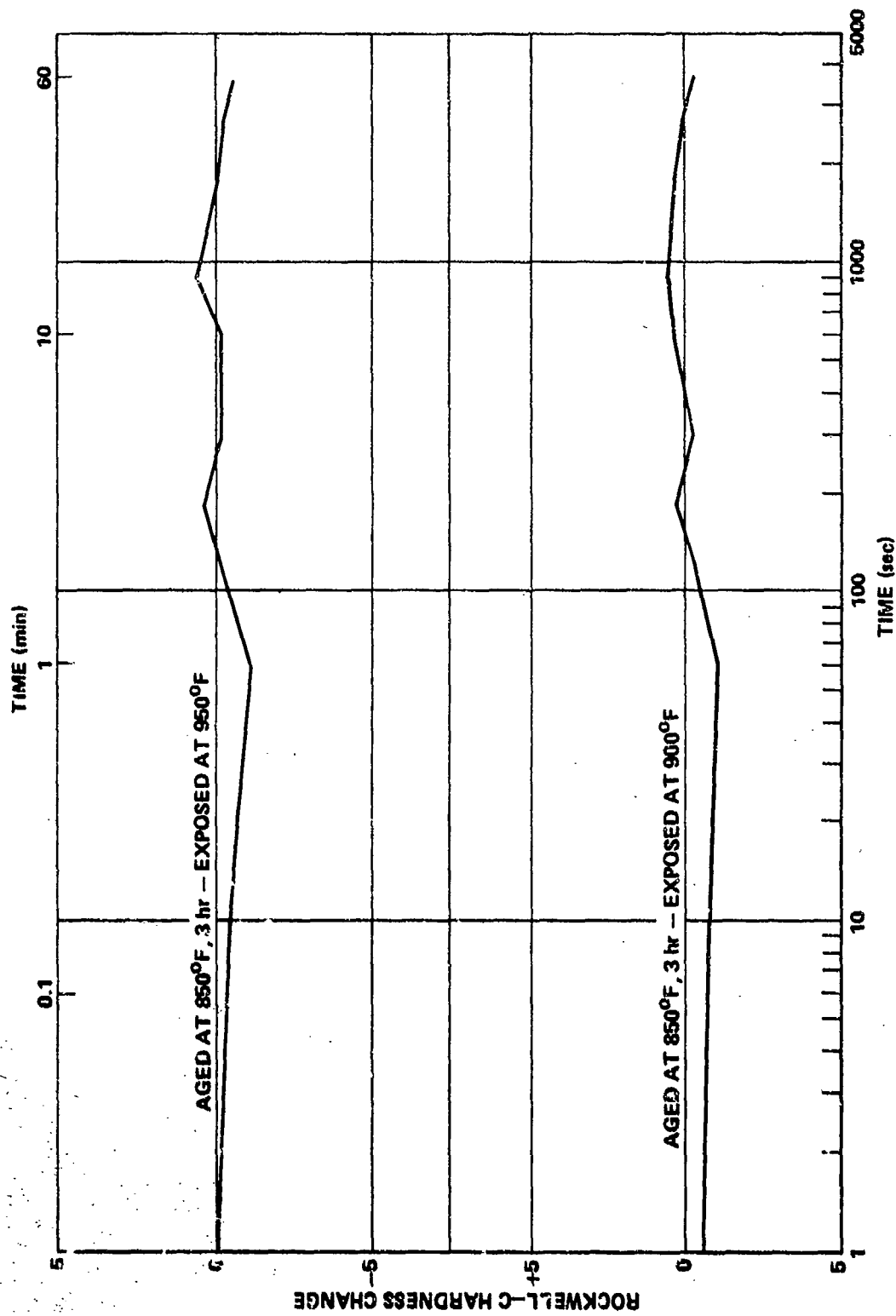


Figure 5. Hardness changes resulting from exposure of 18Ni (300) maraging steel at 900°F and 950°F after aging at 850°F for 3 hours.

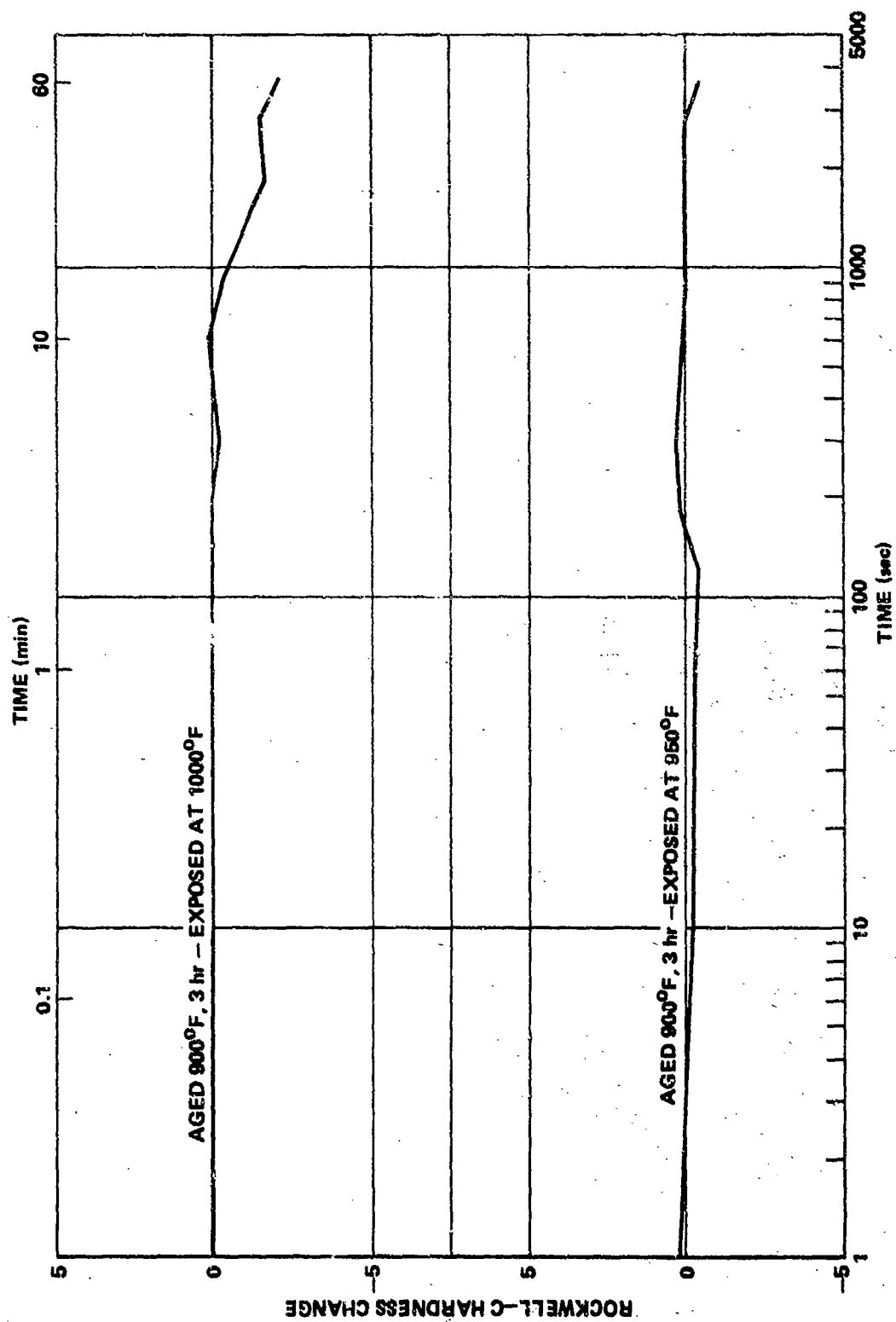


Figure 6. Hardness changes resulting from exposure of 18Ni (300) maraging steel at 950°F and 1000°F after aging at 900°F for hours.

A comparison of the stability of the precipitates from the initial hardening reaction (2-hour age) and those from the second hardening reaction (8-hour age) at 800°F during subsequent exposure at 900°F is shown in Figure 3. The hardness of the samples aged at 800°F for 2 hours decreased 4 points Rockwell C during the first 10 seconds at 900°F and then began to increase so that after approximately 10 minutes the initial aged hardness was recovered. Longer exposures at this temperature result in a further increase in hardness as precipitation of the stable precipitate continues. The hardness of samples aged at 800°F for 8 hours remains relatively stable during a 1-hour exposure at 900°F indicating that the precipitate from the second hardening reaction remains stable during this exposure.

Similar hardness reversions and recoveries are also evident for the initial precipitates formed during aging at 850°F during subsequent exposure at 900°F and 950°F (Figure 4). Samples exposed at these temperatures after aging at 850°F for 3 hours, which corresponds to a point between the two hardening reactions on the 850°F isothermal aging hardness change curve (Figure 1), show an initial hardness reversion of slightly over 1-point Rockwell C. This slight hardness reversion is recovered after approximately 200 seconds at either of these exposure temperatures (Figure 5). The hardness of samples given the standard 3-hour, 900°F aging treatment is stable during exposures of at least 1 hour at 950°F and 10 minutes at 1000°F. After 10 minutes at 1000°F, hardness begins to decrease as overaging occurs (Figure 6).

These observations confirm the metastability of the initial precipitate nucleated during aging at temperatures below 850°F and the stability of the second precipitate nucleated at these temperatures. It is apparent that the strength of material aged for insufficient time to obtain the stable precipitate at lower aging temperatures could be lower for short exposures than for long exposures in the 900° to 1000°F temperature range. It is also possible that a strength reversion caused by the metastable precipitate could also adversely affect the heating rate dependence of this steel in rapid heating tests with concurrent constant loading.

b. Test Specimens and Heat Treatment

Pin-loaded tensile specimens were machined from the mill-annealed sheet with the 0.500 inch wide, 2-inch gage length oriented parallel to the final sheet rolling direction. Since it is well documented [5-7] that aging the 18Ni maraging steels at temperatures below 850°F results in mechanical instability of these steels, the lowest aging temperature considered in this investigation was 850°F. The maraging kinetics and precipitate stability studies previously described indicated that a 3-hour, 850°F aging treatment would be the minimum treatment that could be used with the experimental material to insure development of adequate stability for engineering application and also

allow the retention of some degree of metastability of the microstructure. This aging treatment and the standard 3-hour, 900°F aging treatment were used in this investigation. Both aging treatments resulted in 0.2 percent offset tensile yield strengths of approximately 300,000 psi.

c. Test Apparatus and Test Procedures

All mechanical testing was performed with an MTS closed-loop electrohydraulic testing machine with loading rates and levels under program control. Strain measurements were obtained with a clip-type strain-gage extensometer positioned to record changes in displacement between specimen grips. A 22.5 KVA low-output voltage transformer provided the high electrical current for resistance heating of the test specimens in the heating rate test procedure. An RI Controls three-zone quartz-lamp radiant heating furnace under manual control was used to obtain rapid uniform heating and maintain a uniform isothermal temperature of the test specimens in the short-time elevated temperature tensile test procedure. Both heating methods were adjusted to obtain a uniform temperature within the center 1-inch section of the specimen gage length with temperature variations of less than 15°F during heating and less than 10°F under isothermal conditions. Steep temperature gradients between the isothermal zone and the ends of the specimen gage length restricted plastic deformation of the test specimen to the 1-inch uniform temperature zone.

Temperatures were monitored at 0.5-inch intervals along the 2-inch gage length with 30-gage chromel-alumel thermocouples percussively welded to the test specimen. A drop of ceramic cement shielded the hot junctions of the thermocouples from direct radiation when radiant heating was used. Comparison of the temperature indicated by the surface thermocouples with that indicated by a thermocouple embedded at the center of thickness of a calibration test specimen indicated a through thickness temperature gradient of up to 25°F during radiant heating, which decreased to less than 10°F after a 10-second isothermal soak at temperatures used in this investigation. Output voltages of the thermocouples were recorded as a function of time with appropriate strip-chart recorders in the short-time elevated temperature tensile test procedure. The output voltage of the thermocouple located at the center of the specimen gage length was recorded on the Y-axis of an X-Y recorder with elongation recorded on the X-axis to obtain temperature-elongation curves in the heating rate test procedure.

(1) Heating Rate Test Procedure. The specific heating rate test procedure consisted of applying the required tensile loading within 1 second and initiating resistance heating 2 seconds after the load was applied. Nominal heating rates of 50°, 100°, and 400°F/sec with stress levels of 25, 45, 65, 75, and 85 percent of the room temperature yield strength of the material.

determined in a 4000 psi/sec constant loading rate tensile test, were used in this investigation. Temperature-elongation curves recorded with the X-Y recorder show an initial, approximately linear increase in elongation with increasing temperature due to thermal expansion and decrease in the elastic modulus with increasing temperature. When the temperature reaches a level at which the material can no longer sustain the imposed load without deforming plastically, elongation increases at a more rapid rate with increasing temperature as plastic deformation proceeds to failure.

The temperature at which plastic deformation begins for a given load may be considered as the proportional limit temperature because this temperature on the temperature-elongation curve is analogous to the proportional limit on the stress strain curve. A 0.2-percent offset yield temperature can be determined from the temperature-elongation curve by drawing a line parallel to the linear portion of this curve at an offset equivalent to 0.2 percent strain. This yield temperature is analogous to the yield stress on a stress-strain curve.

It is also possible to obtain an ultimate temperature, which corresponds to the initiation of necking in the test specimen, from the temperature-time curves recorded with strip chart recorders. If a thermocouple junction is located in an area of the specimen experiencing localized necking, a sharp increase in temperature is indicated by this thermocouple due to localized heating as the cross-sectional area of the specimen decreases. Thermocouples positioned outside of the necked area indicated a decrease in heating rate since resistive heating is concentrated in the necked area of the specimen. The temperature at which these discontinuities occur in the temperature-time curves was taken as the ultimate temperature.

The errors associated with the determination of the proportional limit, yield, and ultimate temperatures were related to the difficulty in determining these values from the temperature-elongation and temperature-time curves. It is estimated that the maximum error involved in determining the proportional limit temperature was $\pm 15^{\circ}\text{F}$. The maximum error involved in determining the yield and ultimate temperatures was $\pm 10^{\circ}\text{F}$.

(2) Short-Time Elevated Temperature Tensile Test

Procedure. The specific procedure for the short-time elevated temperature tensile tests consisted of heating the test specimens to the appropriate temperatures within 10 seconds followed by a 10-second isothermal soak at temperature before initiating loading. Loading rates equivalent to constant stressing rates of 4000, 40,000, and 400,000 psi/sec were used at temperatures of 75°, 400°, 600°, 800°, 1000°, and 1200°F. Load-elongation curves were obtained with an X-Y recorder and the proportional limit, 0.2-percent offset yield, and ultimate stresses were determined for each test condition.

Constant loading rates rather than constant strain rates were used in this investigation to obtain strain rate variations during the test that are comparable to those in the heating rate tests. With a constant loading rate, the strain rate remains essentially constant to the onset of plastic deformation and then increases rapidly at an increasing rate determined by the plastic deformation characteristics of the material in the plastic deformation characteristics of the material in the plastic strain range. In the heating rate test procedure, the strain rate is essentially constant during heating until plastic deformation is initiated and then increases in accordance with the creep characteristics of the material. Constant loading rate tests also offer a more realistic simulation of structural loading conditions, because constant loading rates are often encountered in structures whereas strain rates are seldom constant during both elastic and plastic deformation of a structure.

3. Results and Discussion

a. Heating Rate Tests

The effects of applied stress, heating rate, and aging treatment on the proportional limit, yield, and ultimate temperatures of the 18Ni (300) maraging steel used in this investigation are shown in Figures 7 through 9. A linear relation between these temperatures and the logarithm of the heating rate exists for all test conditions. No inversion in heating rate dependence or significant negative heating rate sensitivity of these temperatures was indicated for either aging treatment. The very slight inversion in the heating rate dependence of yield temperature at the 65-percent of room temperature yield stress level for material aged at 850°F is within the temperature measurement scatter and is therefore considered to be insignificant. Heating rate dependence and heating rate sensitivity are generally lower for stresses equivalent to between 45 and 75 percent of the room temperature yield stress. Because the proportional limit, yield, and ultimate temperatures for these stress levels occur between 600°F and 1100°F, it appears that the temperature at which plastic deformation is initiated during the heating rate test procedure determines the heating rate dependence and sensitivity. The two aging treatments did not significantly affect the heating rate dependence and sensitivity.

The fact that aging treatment does not significantly affect the heating rate dependence and sensitivity of this steel even when a metastable precipitate coexists with a stable precipitate, as is the case for the 3-hour, 850°F aging treatment, indicates that precipitate instability is not involved in the heating rate dependence of this steel at temperatures below 1000°F. Since heating rate dependence and sensitivity tend to be low at temperatures between 600°F and 1000°F, it appears that the cause for this behavior may be the same as that for the negative strain rate dependence of an 18Ni (300) maraging steel at 600°F [4].

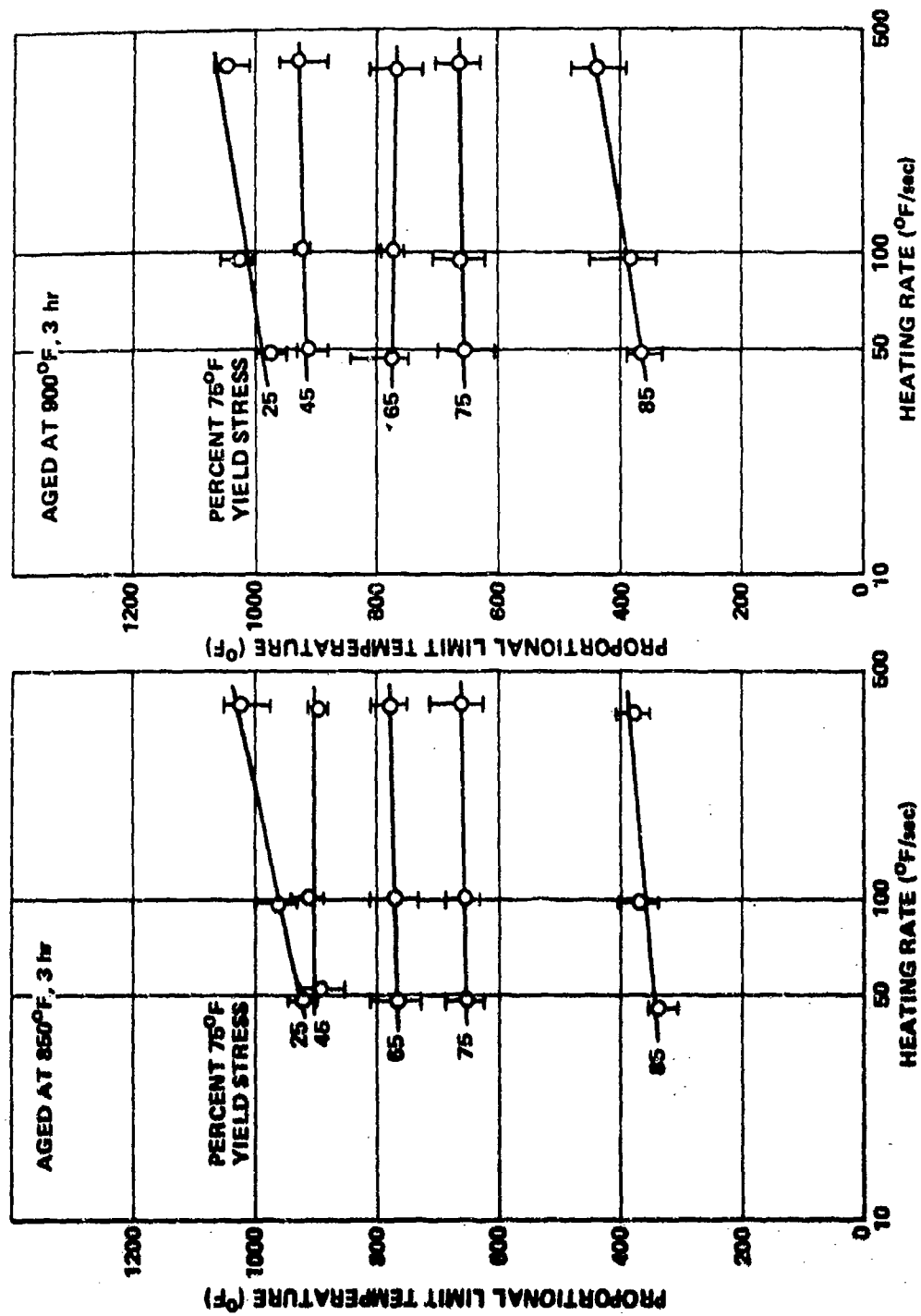


Figure 7. Influence of heating rate on proportional limit temperatures for 18Ni (300) maraging steel.

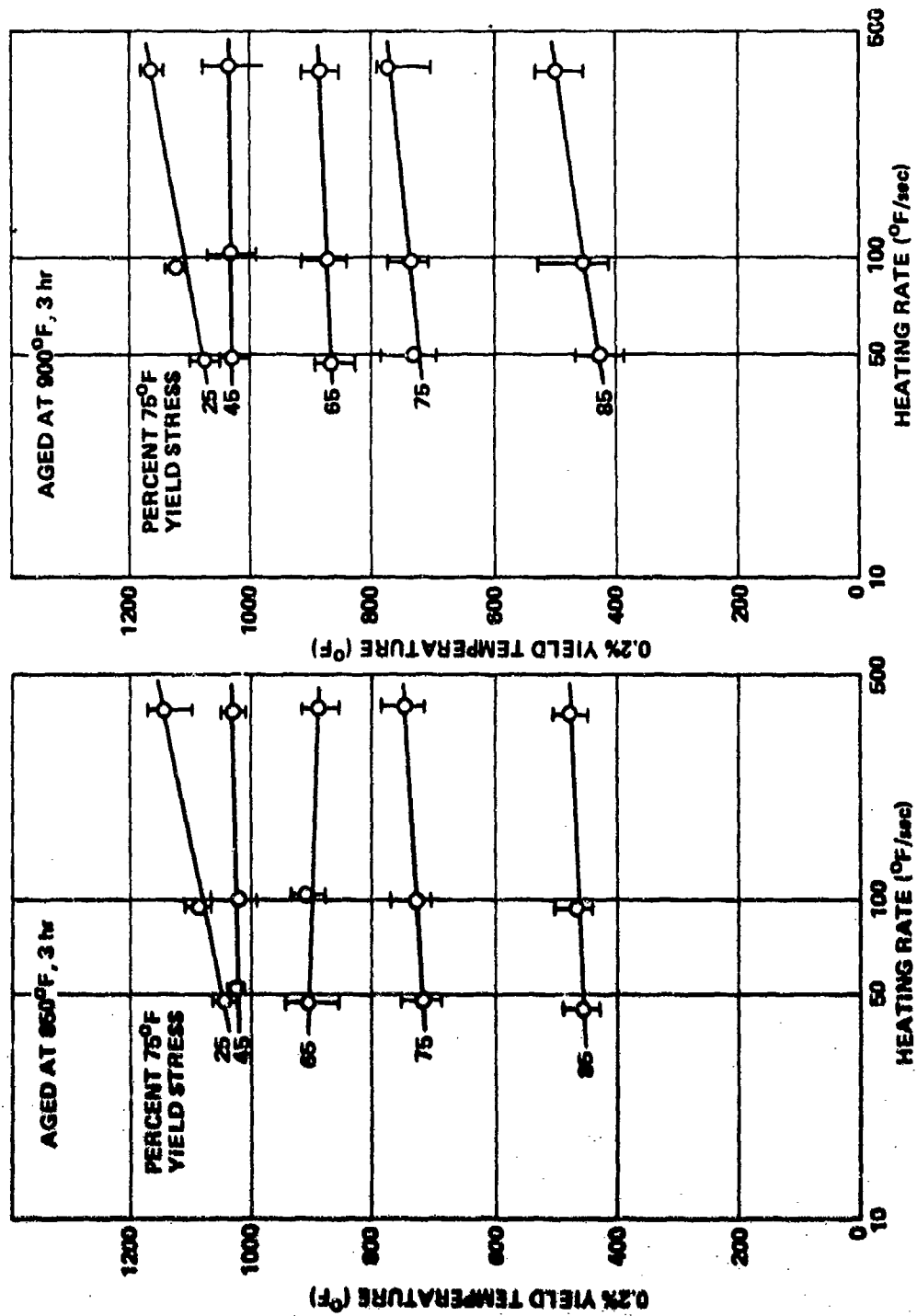


Figure 8. Influence of heating rate on yield temperatures for 18Ni (300) maraging steel.

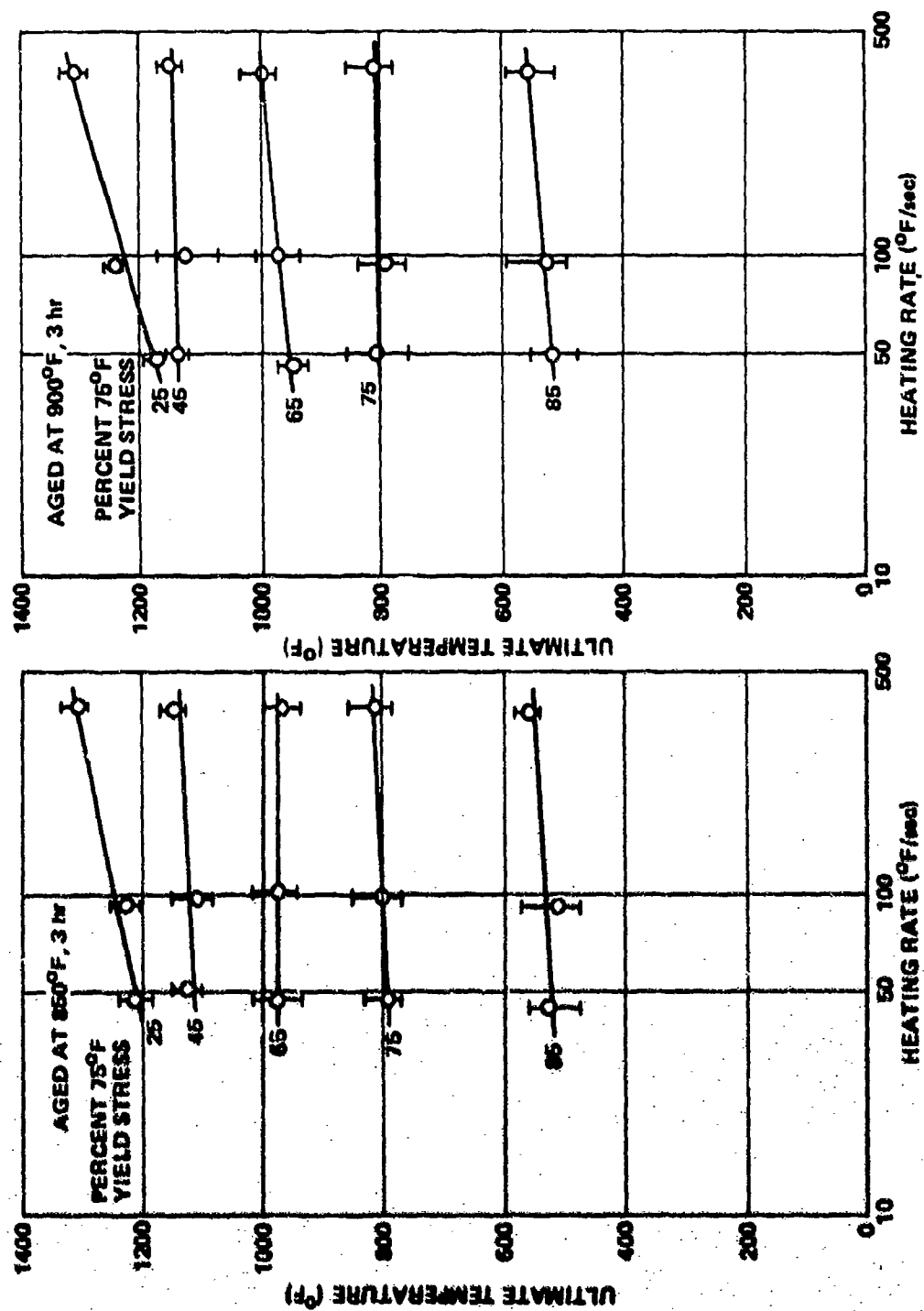


Figure 9. Influence of heating rate on ultimate temperatures for 18Ni (300) maraging steel.

The return of significant heating rate dependence of the proportional, yield, and ultimate temperatures above 1000°F was initially thought to involve reversion of the aged martensite to austenite. Subsequent X-ray diffraction analysis of areas adjacent to fractures obtained at these temperatures did not reveal any significant amount of austenite so the heating rate dependence at these temperatures is apparently related to recovery effects of overaging phenomena.

b. Short-Time Elevated Temperature Tensile Tests

The effects of loading rate and aging treatment on the elevated temperature proportional limit, yield stress, and ultimate stress for this steel are shown in Figures 10 through 12. A linear relation exists between these properties and the logarithm of the loading rate for the 75° to 1200°F temperature range. At room temperature, strength increases with increasing loading rates. As the test temperature is increased through the 400° to 600°F temperature range, loading rate sensitivity decreases resulting in a transition from a positive strength dependence on loading rate to a negative dependence with strength decreasing with increasing loading rates. These results are consistent with those in elastic strain rate (constant loading tests reported by Kendall [4] on 18Ni (300) maraging steel from room temperature to 600°F.

An inverse strength dependence on loading rate was evident throughout the 600° to 1000°F temperature range, while loading rate sensitivity remained essentially constant. As the temperature was increased above 1000°F, the loading rate sensitivity increased and the loading rate dependence of strength became positive. This inversion in loading rate sensitivity is apparently related to overaging phenomena including the reversion of aged martensite to austenite. X-ray diffraction analysis of areas adjacent to fractures obtained in tests performed at 1200°F indicated a significant increase in austenite occurred during these tests.

c. Comparison of Yield and Ultimate Stresses Obtained in the Two Test Procedures

Yield stresses, defined as those stresses at which yield temperatures occur in the heating rate test procedure, are compared with yield stresses obtained in the short-time elevated temperature tensile tests in Figures 13 and 14 for the 850°F and 900°F aging treatments. Corresponding ultimate stresses are compared in Figures 15 and 16.

It is apparent that at temperatures above 600°F, yield stresses obtained in the heating rate test procedure are significantly lower than those obtained in the tensile tests for both aging treatments. The

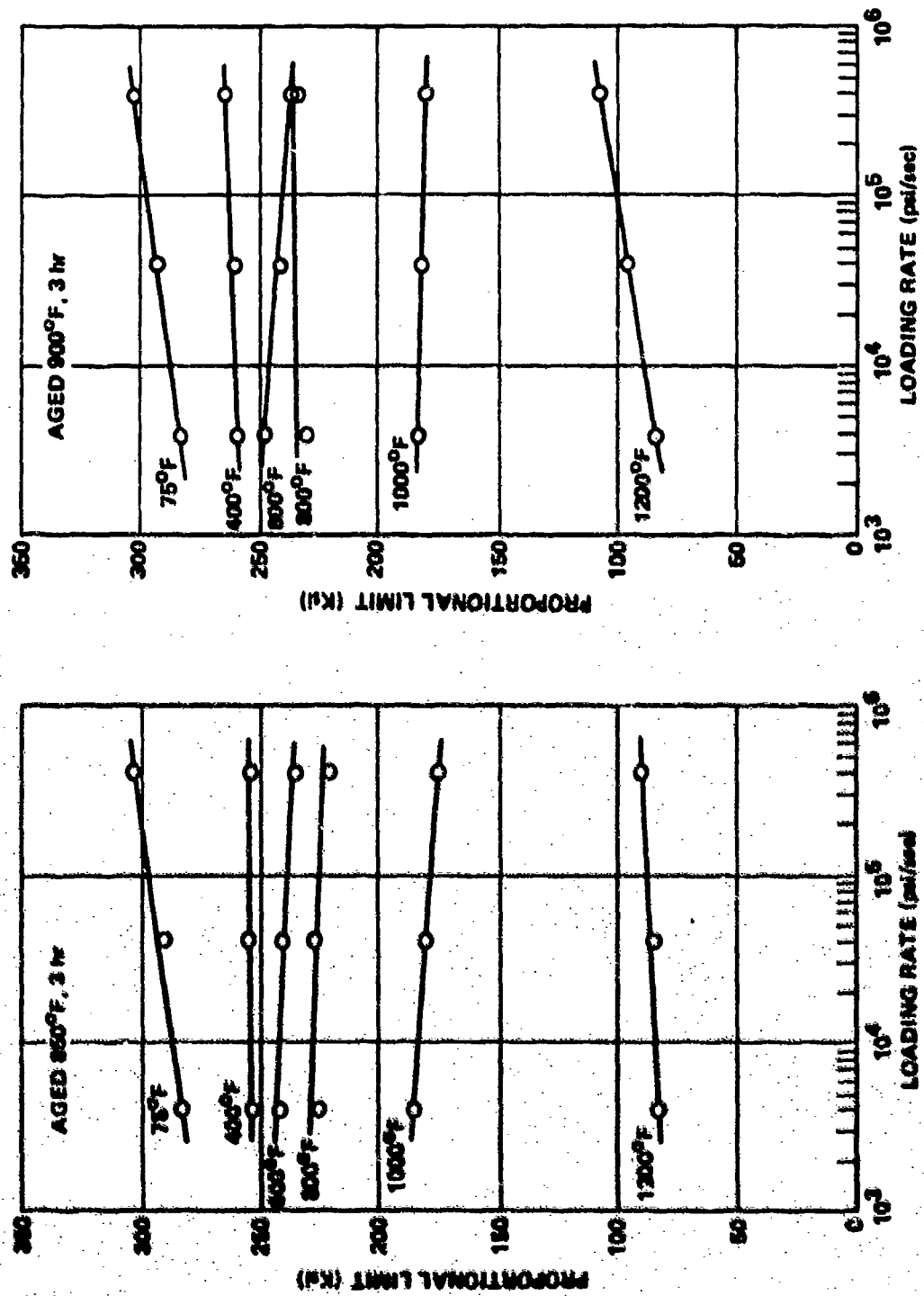


Figure 10. Influence of loading rate on proportional limit of 18Ni (300) maraging steel.

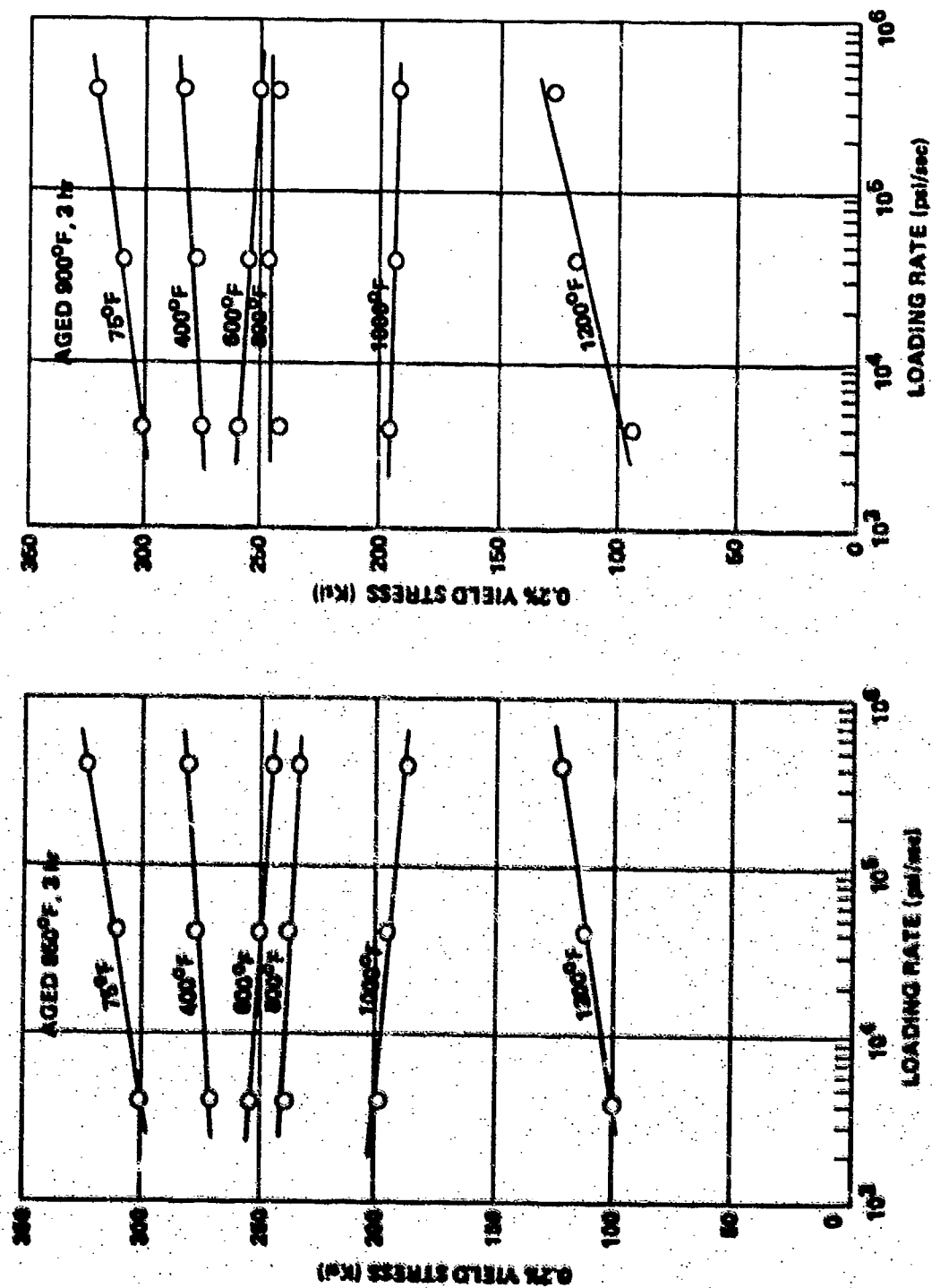


Figure 11. Influence of loading rate on yield stress of 18Ni (300) maraging steel.

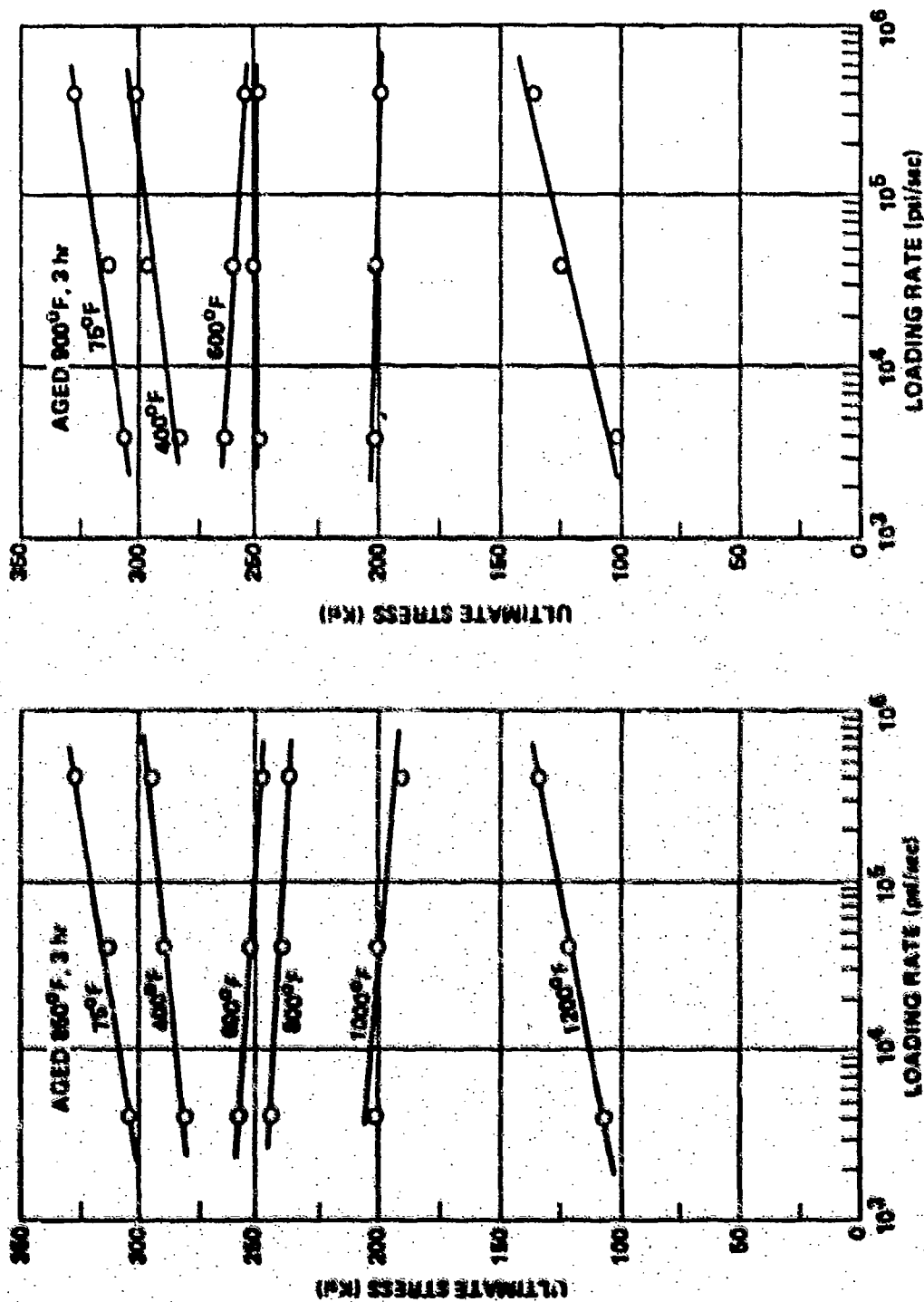


Figure 12. Influence of loading rate on ultimate stress of 18Ni (300) maraging steel.

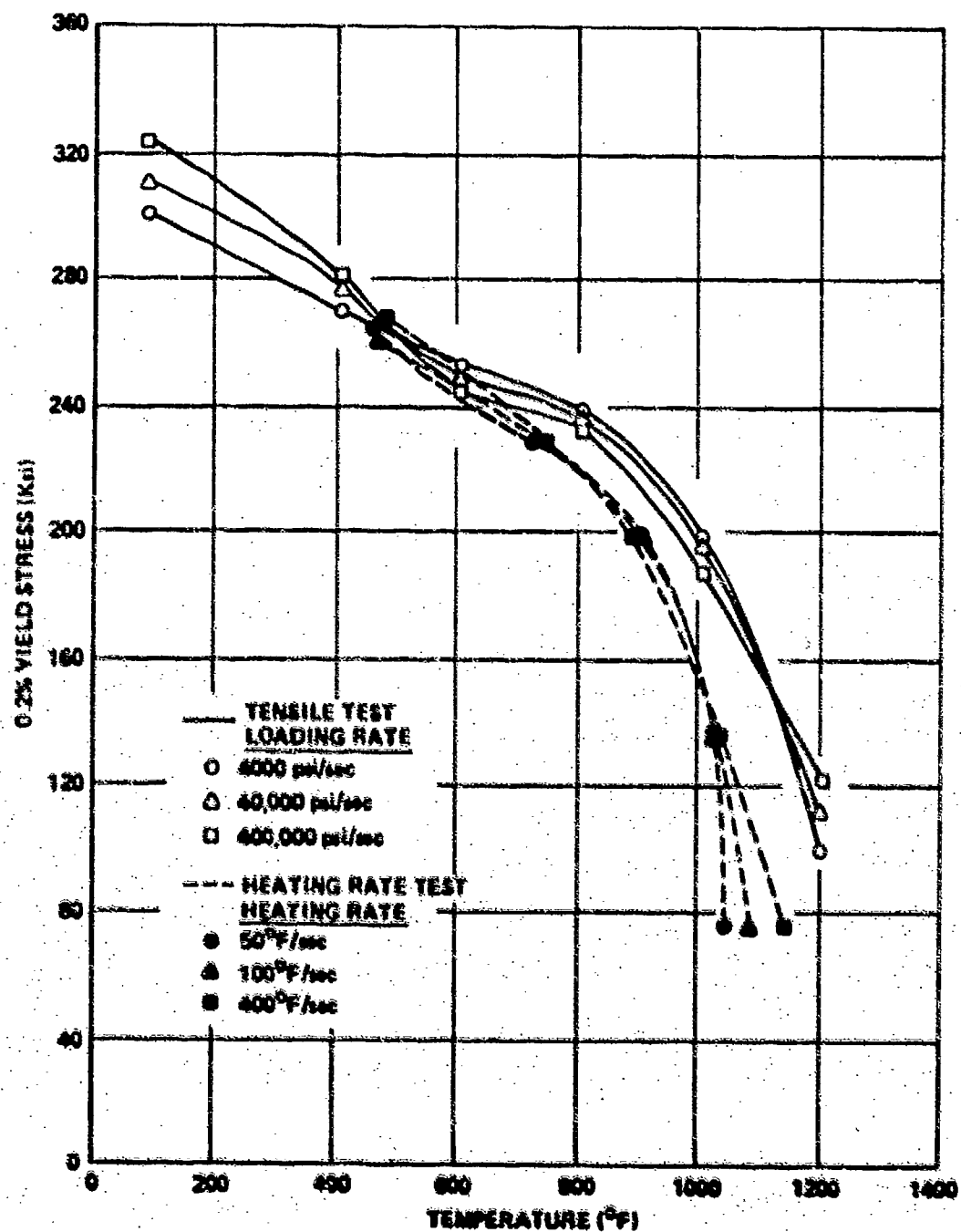


Figure 13. Comparison of yield stresses obtained in tensile tests and heating rate tests on 18Ni (300) maraging steel aged at 850°F for 5 hours.

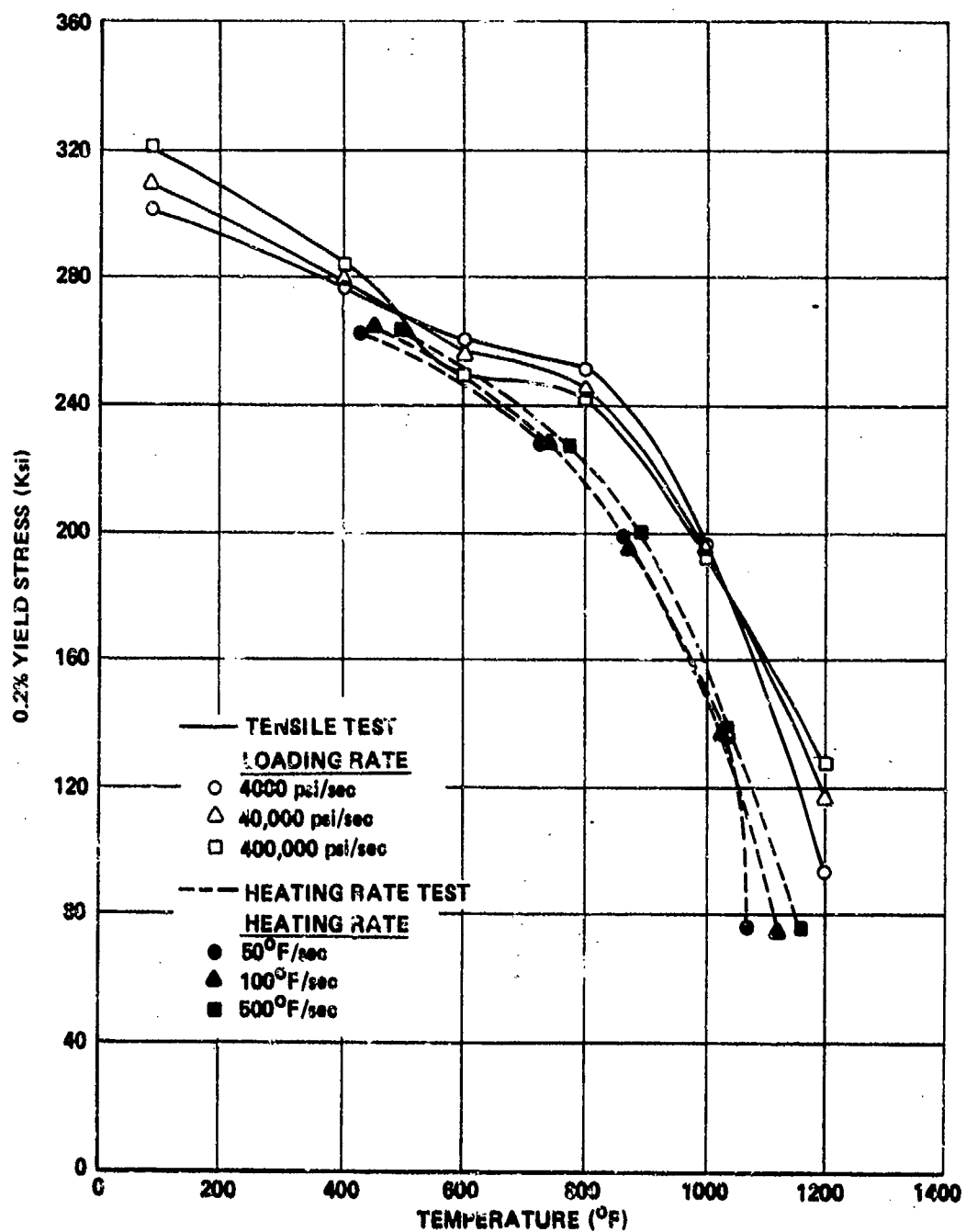


Figure 14. Comparison of yield stresses obtained tensile tests and heating rate tests on 18Ni (300) maraging steel aged at 900°F for 3 hours.

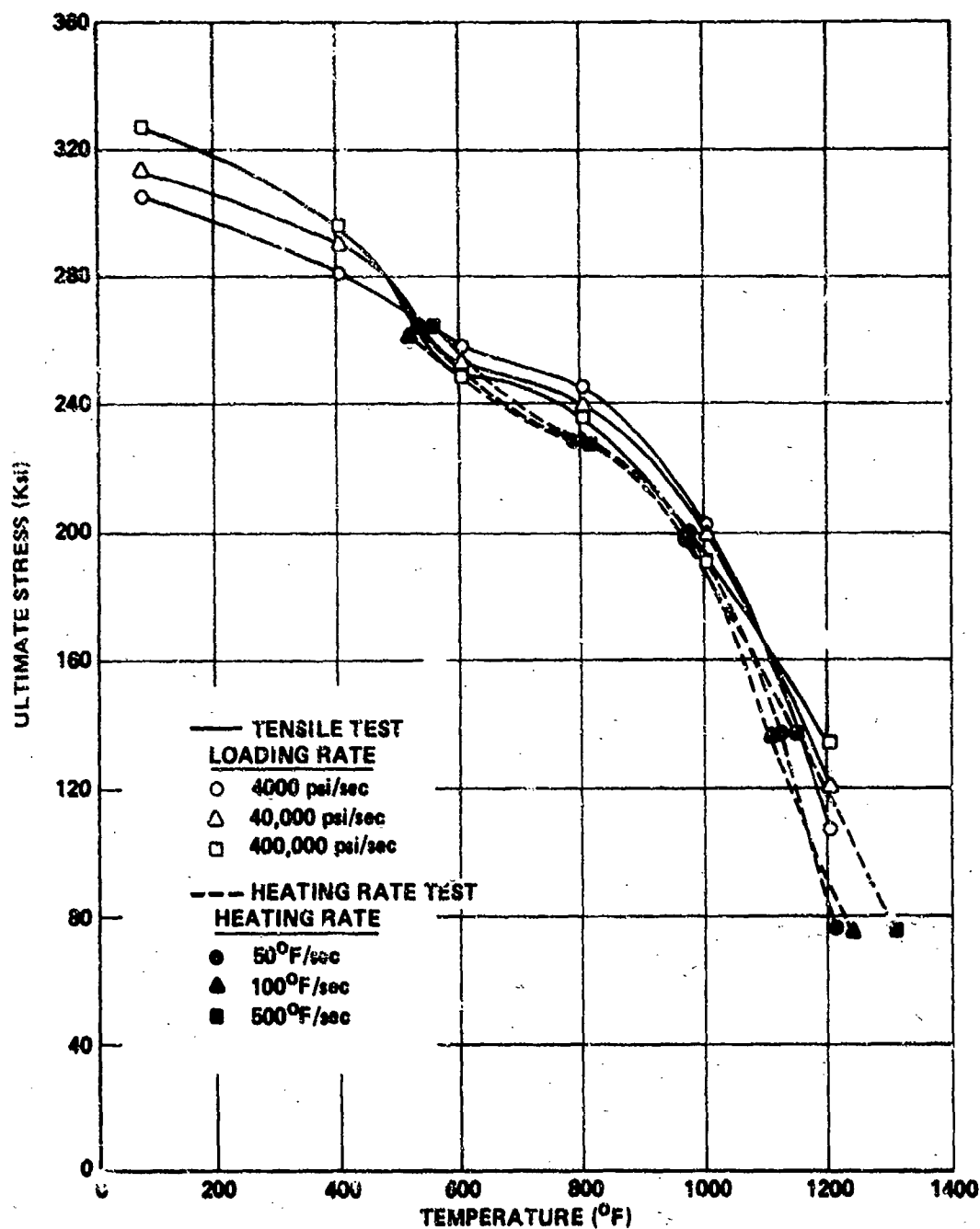


Figure 15. Comparison of ultimate stresses obtained in tensile tests and heating rate tests on 18Ni (300) maraging steel aged at 850°F for 3 hours.

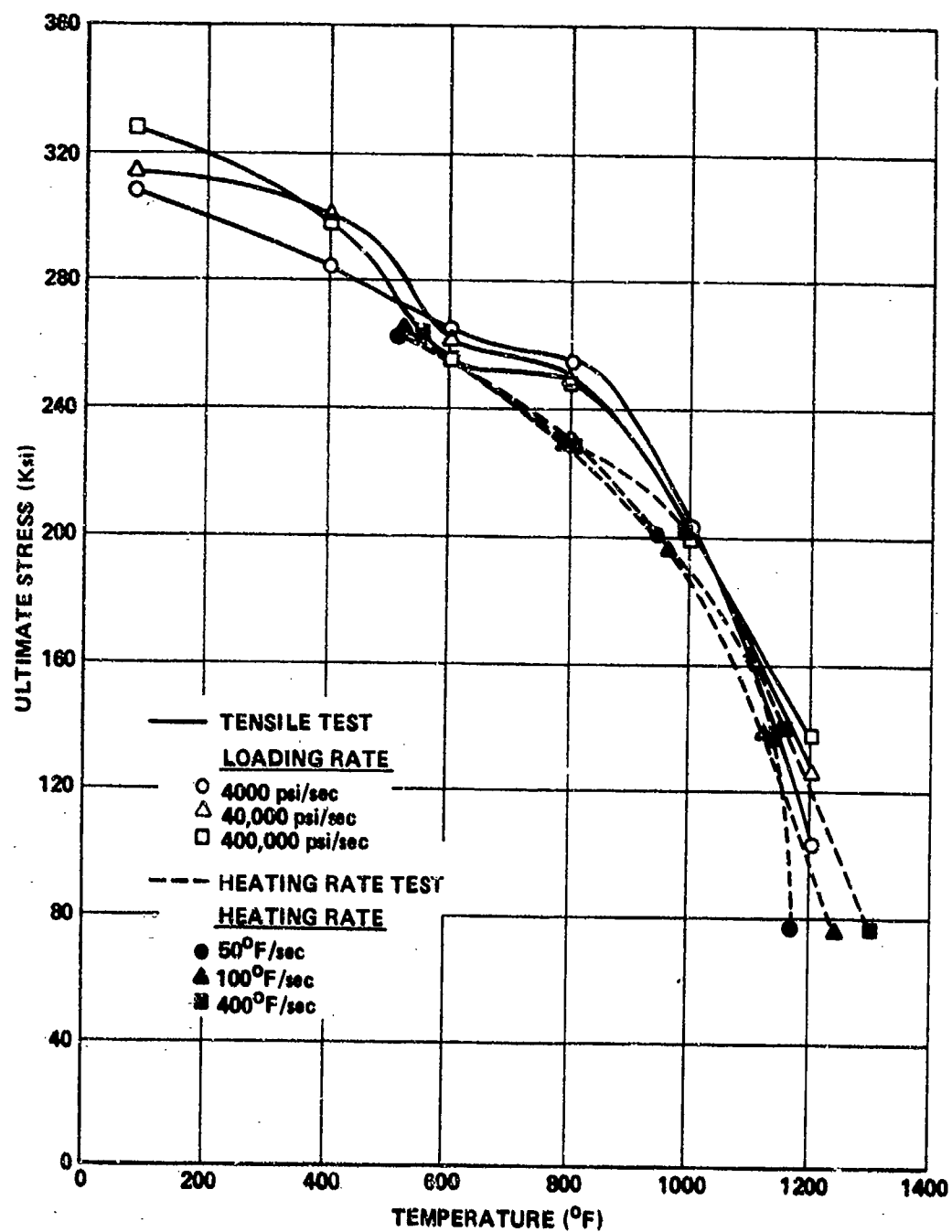


Figure 16. Comparison of ultimate stresses obtained in tensile tests and heating rate tests on 18Ni (300) maraging steel aged at 900°F for 3 hours.

difference between the respective yield stresses becomes greater with increasing temperature, with the difference increasing from approximately 10,000 psi at 700°F to 40,000 psi at 1000°F. Ultimate stresses obtained in the heating rate tests were generally lower than those obtained in the tensile tests at temperatures above 600°F. The most significant difference in the ultimate stresses obtained in the two test procedures occurred in the 700° to 900°F temperature range. Between 900°F and 1000°F for the 850°F aging treatment and between 1000°F and 1100°F for the 900°F aging treatment, the difference between the ultimate stresses obtained in the two test procedures decreases while the difference between the yield stresses increases significantly, indicating that creep is occurring in the heating rate test procedure in these temperature ranges.

Aging treatments used in this investigation did not significantly affect the yield stresses of this steel in the tensile test procedure. In the 600° to 900°F temperature range, the yield stresses of material aged at 850°F were slightly lower, generally less than 15,000 psi, than those of material aged at 900°F. This behavior is related to the slightly increased elevated temperature stability derived from the higher temperature aging treatment. The aging treatments had even less influence on the results of the heating rate tests with the yield stress-temperature curves being essentially identical for the two aging treatments. The 900°F aging treatment did increase resistance to creep to some extent in the 900° to 1100°F temperature range.

d. Comparison of Load-Carrying Ability in the Two Test Procedures

Comparisons of the load-carrying ability of this steel for the two test procedures are shown in plots of time to the yield stress and to the ultimate stresses at selected temperatures. Comparisons for the yield stresses at temperatures of 400°F, 600°F, 800°F, and 1000°F for the two aging treatments are shown in Figures 17 and 18. Similar comparisons for the ultimate stresses are shown in Figures 19 and 20. The time scale in these figures refers to the elapsed time between initiation of loading and attaining the yield or ultimate stress in the tensile test procedure. Times to yield or ultimate stress for the heating rate test procedure was taken as the elapsed time from initiation of heating to reaching the yield or ultimate temperature.

The procedure for obtaining the load-carrying ability for the tensile test procedure consisted of determining the time required to attain the yield or ultimate stress for each loading rate and test temperature. These data were plotted as yield stress or ultimate stress as a function of time for temperatures of 400°F, 600°F, 800°F, and 1000°F. Graphical cross-plotting techniques were used to obtain the load-carrying ability for the heating rate test procedure for comparison with data from the tensile tests. The heating rate test data were first

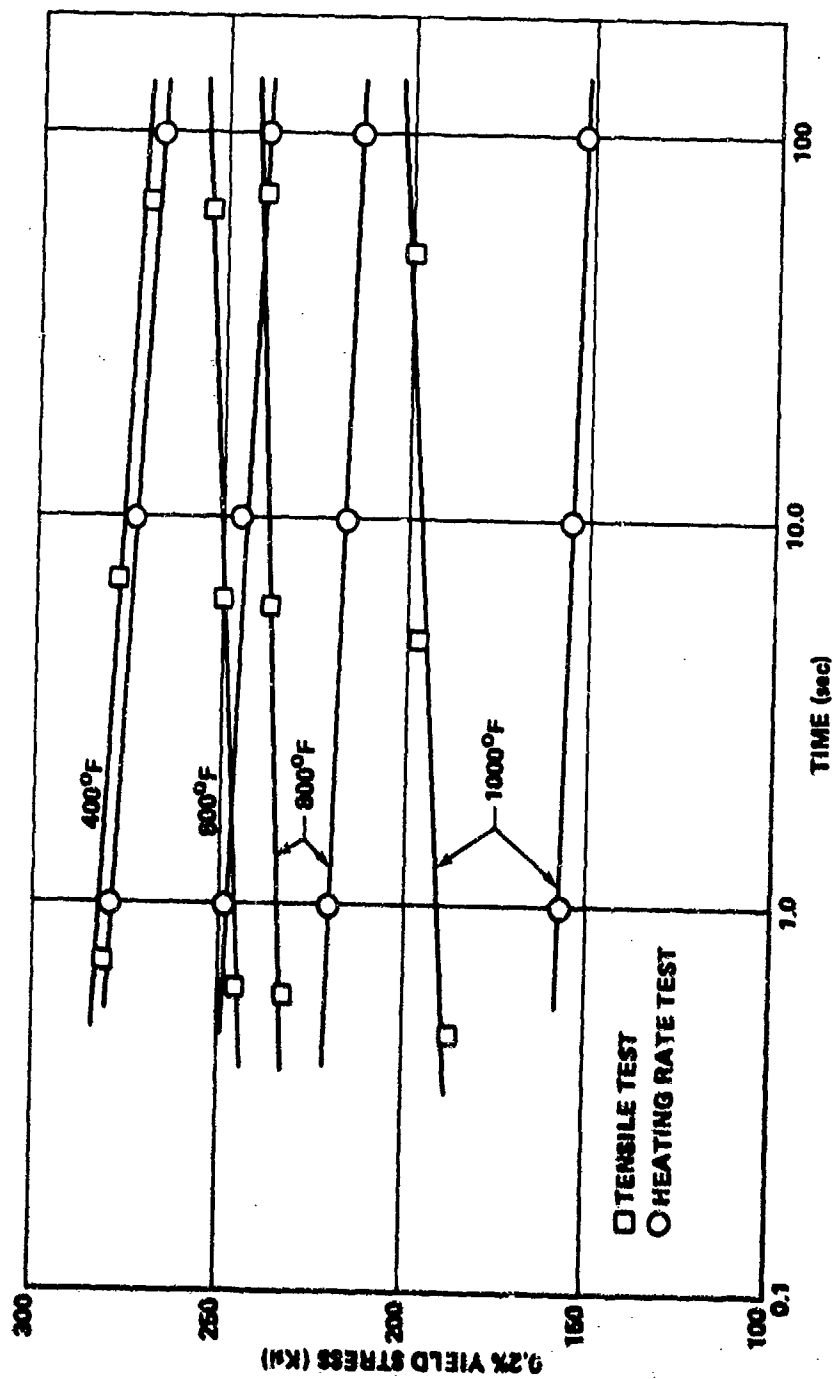


Figure 17. Comparison of the load-carrying ability of 18Ni (300) maraging steel aged at 850°F for 3 hours based on yield stress in tensile tests and heating rate tests.

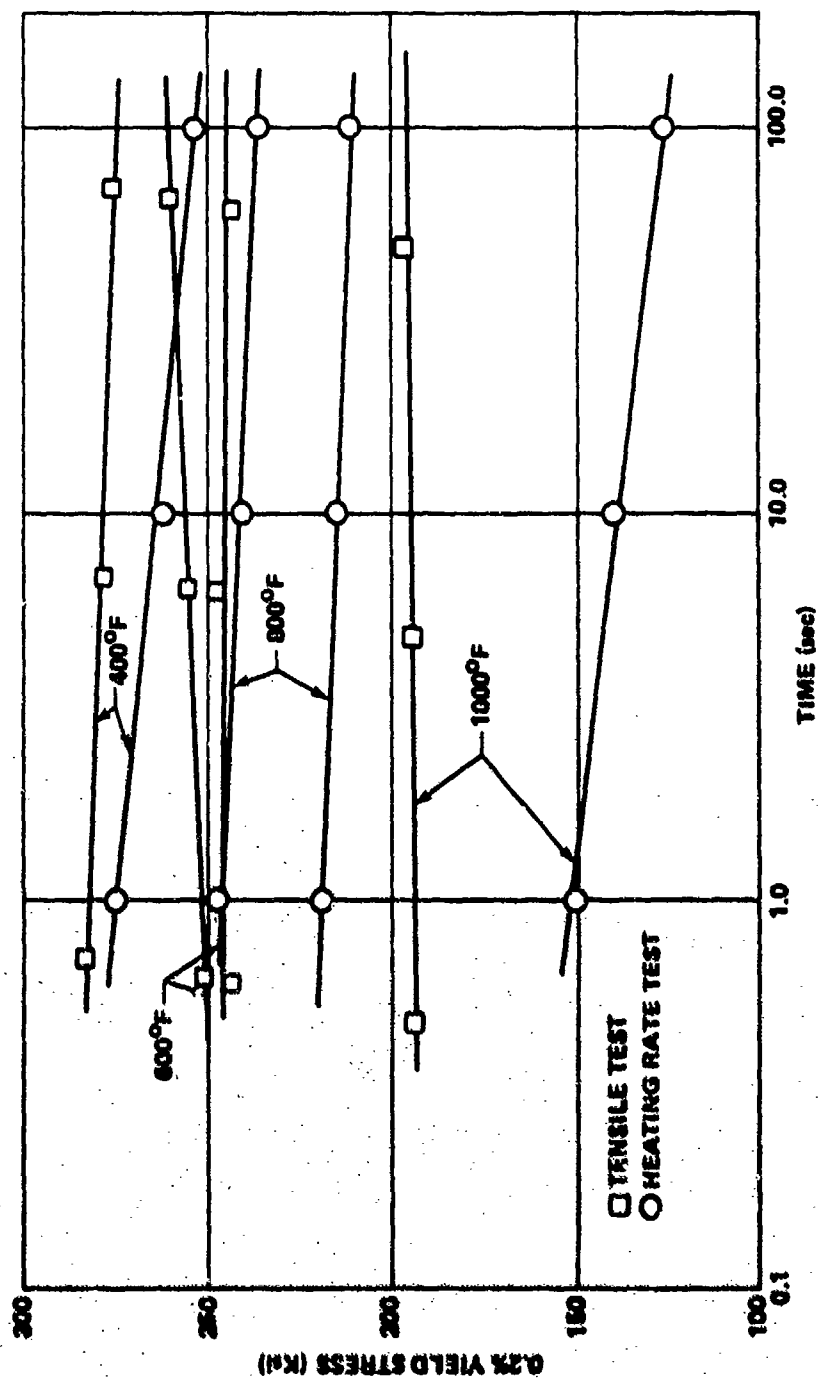


Figure 18. Comparison of the load-carrying ability of 18Ni (300) maraging steel aged at 900°F for 3 hours based on yield stress in tensile tests and heating rate tests.

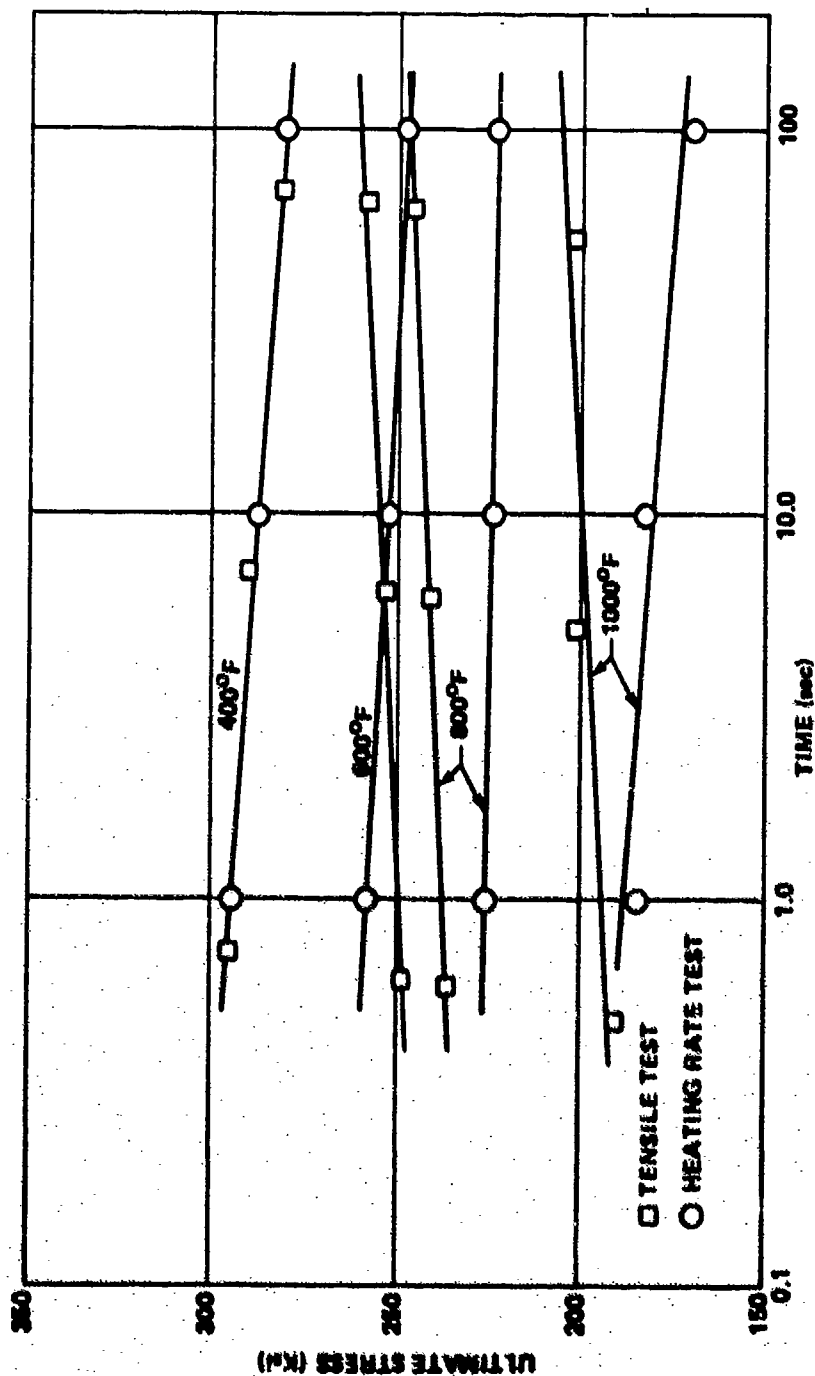


Figure 19. Comparison of the load-carrying ability of 18Ni (300) maraging steel aged at 850°F for 3 hours based on ultimate stress in tensile tests and heating rate tests.

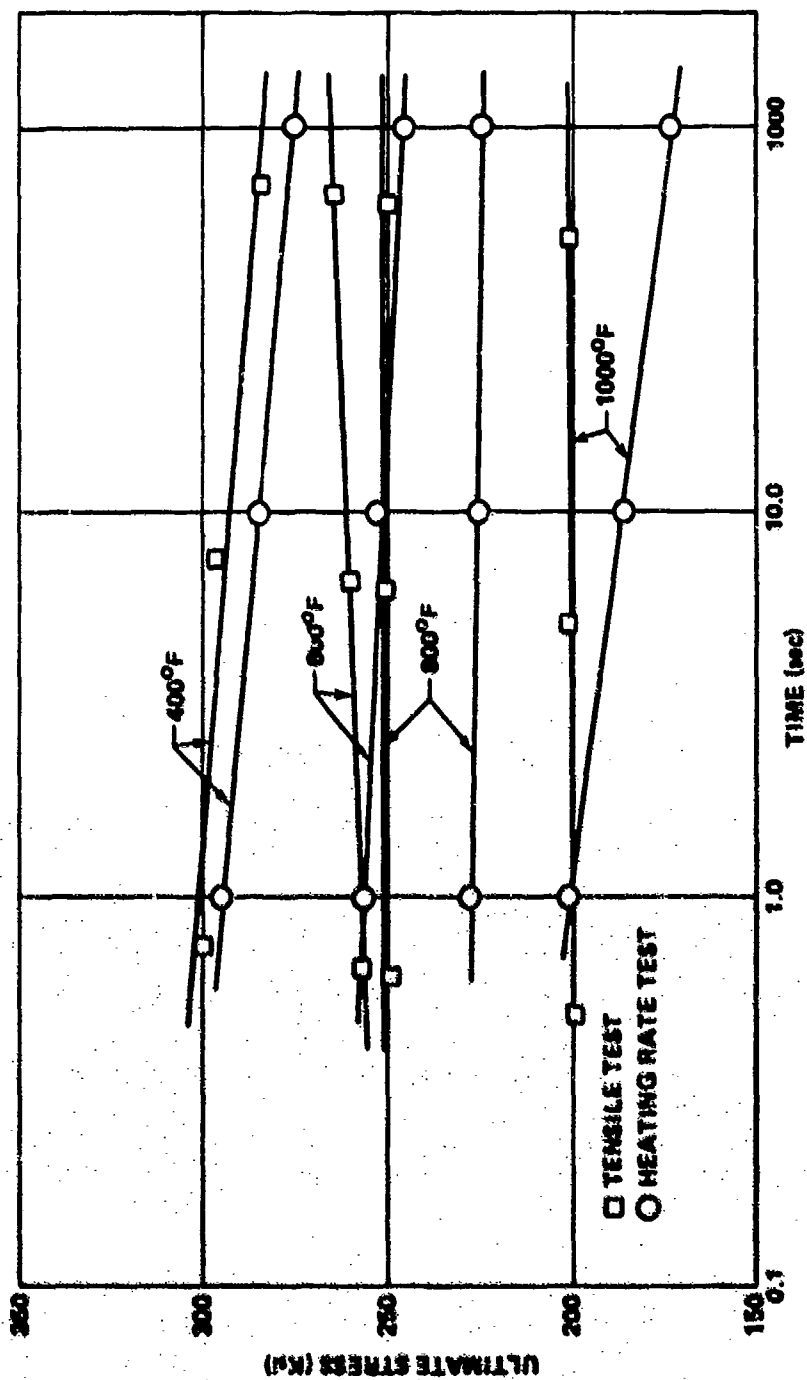


Figure 20. Comparison of the load-carrying ability of 18Ni (300) maraging steel aged at 900°F for 3 hours based on ultimate stress in tensile tests and heating rate tests.

plotted in the form of constant stress curves showing the yield and ultimate stresses as a function of heating time. Values obtained from the constant-time intercepts of these curves were used to cross plot stress as a function of temperature for times of 1, 10, and 100 seconds. The stress-time intercepts on these curves were used to plot stress-time curves for temperatures of 400°F, 600°F, 800°F, and 1000°F for comparison with those derived from the tensile test data.

Load-carrying ability for the heating rate test procedure decreases with elapsed time at all temperatures, while the load-carrying ability for the tensile test procedure decreases with time at 400°F, but increases with elapsed time at 600°F, 800°F, and 1000°F. Load-carrying ability is generally greater for the tensile test procedure than for the heating rate test procedure with the exception that the load-carrying ability based on the ultimate stress is greater for times of less than 1 second in the heating rate test procedure at 600°F and 1000°F for the 900°F aging treatment and that the load-carrying ability based on both the yield and ultimate stress is greater for times of less than 2 and 8 seconds respectively in the heating rate test procedure at 600°F. The load-carrying ability determined from the tensile test procedure for 800°F is higher than that for the heating rate test procedure for 600°F for times greater than 60 seconds for the 850°F aging treatment and for shorter times for the 900°F aging treatment. These results indicate that the load-carrying ability of this steel under a given sequence of heating and loading cannot be predicted from tests conducted under a different sequence of heating and loading even though the times and temperatures under heating and loading are the same.

c. Strain Rate Sensitivity and Strain Rate Inversion

Strain rate sensitivity of a material denotes variation of mechanical properties with the rate of deformation. It is defined quantitatively as the ratio of the increments of the logarithm of stress and the logarithm of strain rate at a given strain. Strain rate sensitivity can be studied quantitatively by many types of mechanical tests, but the easiest tests to interpret are those in which a steady-state condition of plastic flow is disturbed by a sudden change in load or strain rate so that the resulting stress change is due entirely to the rate change and not partly due to strain hardening. When this type of experimental data is not available, it is possible to obtain an apparent strain rate sensitivity by cross plotting strength values for a particular plastic strain obtained for various strain rates.

The apparent strain rate sensitivity determined in this manner depends on the nature of metallurgical stability affecting the strain hardening characteristics of the material. Under conditions where recovery occurs, the apparent strain rate sensitivity is higher than the true strain rate sensitivity. When strain aging occurs, the apparent strain rate sensitivity is lower than the true strain rate sensitivity.

The apparent strain rate sensitivity of a material is a highly sensitive measure of the metallurgical stability of the material and therefore would appear to be the more important parameter for the analysis of the rate sensitive mechanical behavior of a material in engineering applications.

To obtain the apparent strain rate sensitivity of 18Ni (300) maraging steel from the constant loading rate tensile test data at the various test temperatures, the average strain rate between 0.05 and 0.2 percent offset plastic strain was taken as the plastic strain rate for each test condition. The portions of the stress-strain curves involved in the plastic strain rate determinations were essentially linear over this strain range. The 0.2 percent offset yield stresses plotted as a function of the logarithm of the plastic strain rates for the test temperatures and aging treatments used in this investigation are shown in Figure 21. A linear relation between yield stress and the logarithm of plastic strain rate exists for all test conditions. This is the same relationship that has been observed at low temperatures for unaged 18Ni maraging steel martensite by Leslie and Sober [13] and at temperatures from room temperature to 600°F by Kendall [4] for aged 18Ni (300) maraging steel. These plots show essentially the same strain rate dependence characteristics as the elastic strain rate data of Kendall for the room temperature to 600°F range and are essentially analogous to the loading rate plots for temperatures to 1200°F described previously in this report.

The apparent strain rate sensitivity defined as the logarithm of the ratio of the yield stresses for the highest and lowest strain rates divided by the logarithm of the ratio of the corresponding plastic strain rates was determined for each test temperature. The effect of temperature on the apparent strain rate sensitivity of this steel is shown in Figure 22. The decrease in the apparent strain rate sensitivity of this steel between 400°F and 600°F along with the negative values for this parameter in the 600° to 1000°F range results in an inversion in the strain rate dependence of yield stress in this temperature range. These factors are indicative of dynamic strain aging in this temperature range.

f. Dynamic Strain Aging

Strain aging is generally defined as a time-dependent change in properties which results from a sequence of plastic straining and aging. Since strain aging can be explained in terms of dislocation pinning by diffusing impurity atoms [12], the kinetics of the process should depend largely on the diffusion rates of the impurity atoms. Dynamic strain aging occurs when the diffusion rate of dissolved impurity atoms becomes high enough so that pinning of mobile dislocations occurs during plastic deformation. As a result, strain aging phenomena may occur simultaneously with deformation at appropriate temperatures.

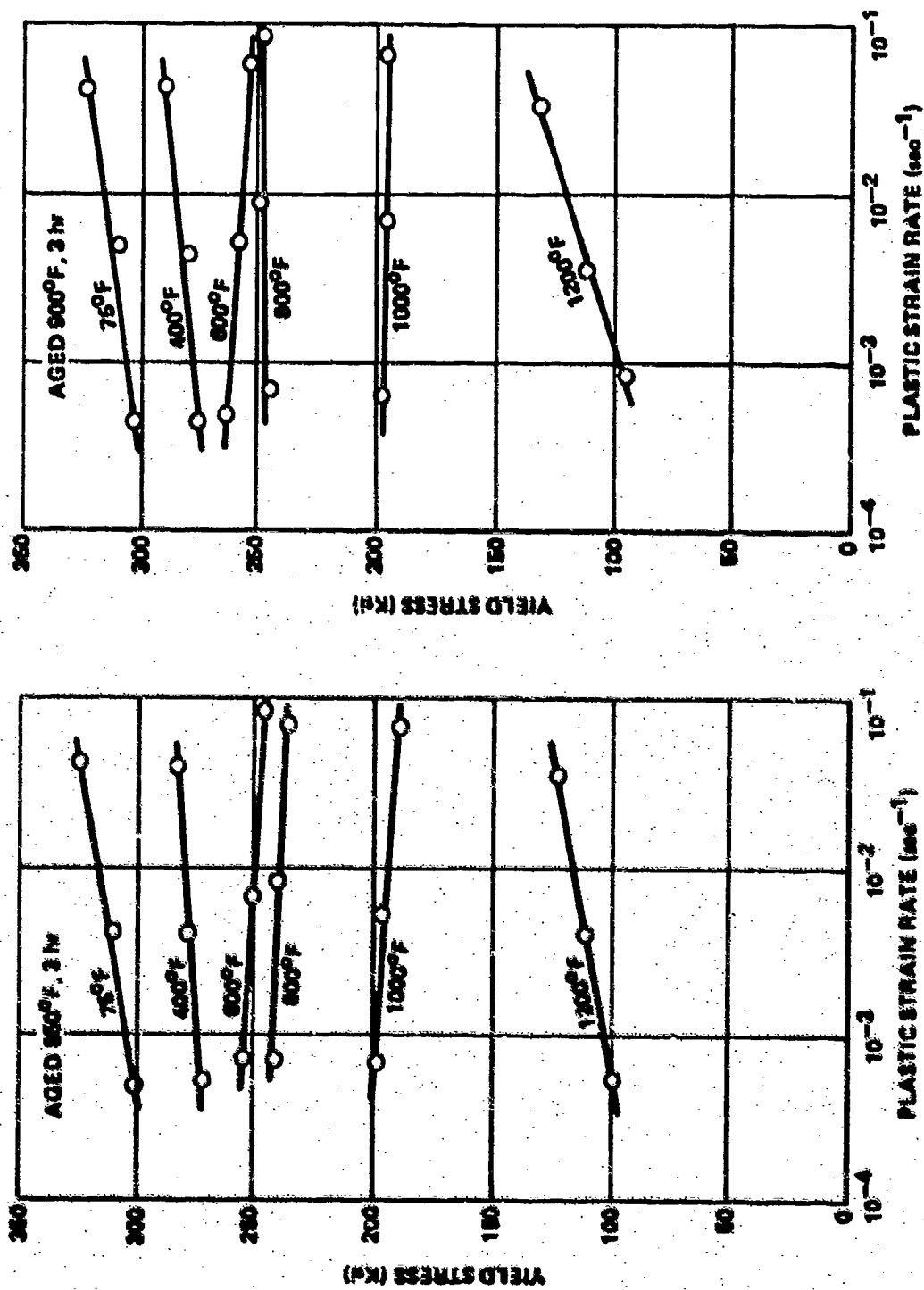


Figure 21. Influence of plastic strain rate on the yield strength of 18Ni (300) maraging steel.

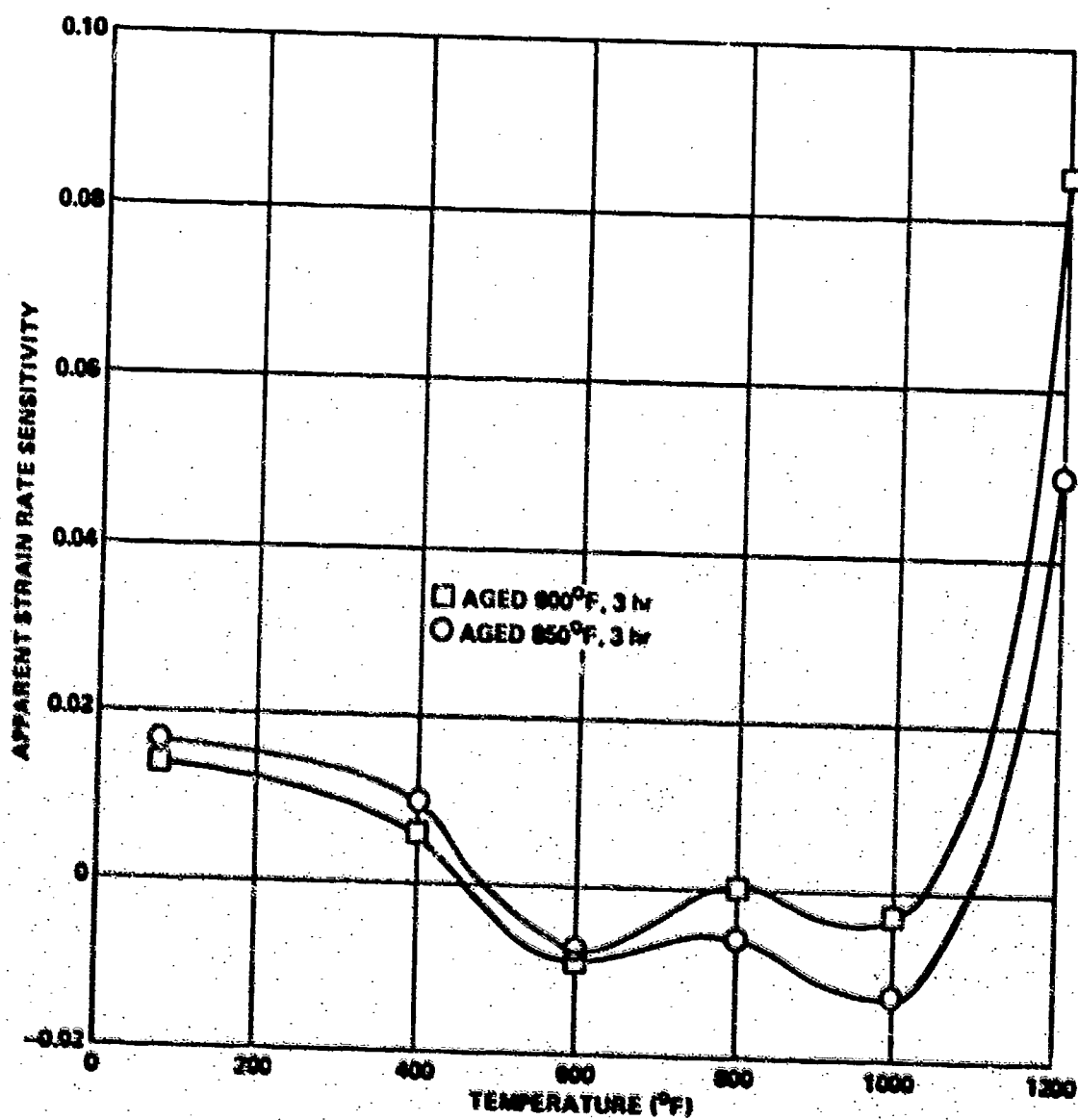


Figure 22. Temperature dependence of the apparent strain rate sensitivity of 18Ni (300) maraging steel.

Dynamic strain aging manifests itself in one or more aspects including discontinuous yielding, abnormally low strain rate sensitivity, abnormal work hardening rates, peaks or plateaus in flow stress-temperature curves, and ductility minima in most metallic materials.

Owen and Roberts [14] have observed that interstitial carbon atoms promote dynamic strain aging in iron-nickel martensites containing at least 18-weight percent nickel and more than 0.003-weight percent carbon. Steigerwald and Hanna [15] have also presented evidence that dynamic strain aging occurs in 18Ni maraging steel and several other high-strength steels in the 300° to 800°F temperature range. Dynamic strain aging in both the iron-nickel martensite and the aged martensite of the 18Ni maraging steel have been attributed to dynamic Cottrell interactions [16].

Dynamic strain aging resulting from Cottrell dislocation pinning by diffusing interstitial atoms can also account for the anomalous strain rate sensitivity and inverse strain rate effects observed in this investigation. When the strain rate is increased in the temperature range in which dynamic strain aging can occur, less time is available for diffusion of impurity atoms to occur during deformation thus reducing their effectiveness in pinning dislocations. This results in a decrease in strain-hardening which allows yielding to occur at lower stresses in this temperature range. The effect of dynamic strain aging on the mechanical behavior of 18Ni (300) maraging steel depends upon the temperature within the 500° to 1000°F temperature range, the strain rate, and the amount of impurity atoms available in the martensite matrix.

Gillis [17] has proposed that the impurity diffusion mechanism cannot explain the physical mechanism underlying inverse strain rate effects. He has proposed that the Johnston-Gilman theory of yielding [18], which predicts that flow stress at a given strain must increase with increasing strain rate, in conjunction with stress induced structural instability theory of Morris [19], which predicts that solute precipitation occurs prior to yielding with the amount of precipitation depending inversely on the strain rate, provides the physical basis for inverse strain rate effects. This theory accounts for the change from normal strain rate dependence of strength observed at low and very high strain rates to inverse strain rate dependence at intermediate strain rates observed for a number of materials.

Steigerwald and Hanna [15] have proposed that if precipitation occurs during tensile tests in the strain aging temperature range, the precipitate should also influence the mechanical properties at other temperatures following prestraining in this temperature range. An attempt was made to test this hypothesis for the dynamic strain aging of this steel. Duplicate test specimens were prestrained 0.5 percent at 600°F at each of the three loading rates used in this investigation. The specimens were rapidly cooled in a water spray and tested immediately

upon reaching room temperature at a loading rate corresponding to 4000 psi/sec. No significant changes in yield or ultimate stresses were observed after these prestraining treatments.

While the results of this experiment do not provide conclusive evidence that precipitation does not occur during strain aging, they do indicate that any precipitation that may have occurred in these short-time tests does not significantly affect the subsequent room temperature mechanical behavior of this steel. The results of stress aging investigations of Harrington [17] and Wright [18] appear to support the theory that precipitation occurs during deformation of maraging steel in the strain aging temperature range. They found that significant increases in the room temperature yield stress of both 18Ni (250) and 18Ni (300) maraging steel can be obtained after prestraining under constant stress for times of 15 minutes to 4 hours in the 600° to 900°F temperature range. The results of these stress aging studies would appear to support the theory that significant solute precipitation occurs in these steels during deformation in the strain aging temperature range, provided deformation rates are low and deformation times are sufficiently long.

The results of this investigation indicate that the elevated temperature dynamic strain aging effects in 18Ni (300) maraging steel observed in short-time test procedures are a unique function of the temperature range in which sufficient interstitial mobility exists. This would indicate that the impurity diffusion theory offers the best description for the strain rate sensitivity and inverse strain rate effects observed during short-time elevated temperature tests on this steel. It is also apparent that the dynamic strain aging effects are uniquely associated with the martensite matrix and do not involve any instability of the intermetallic precipitates formed during the aging of this steel for 3 hours at either 850°F or 900°F.

4. Conclusions

A linear relation exists between the strength of 18Ni (300) maraging steel and the logarithm of the heating rate and loading rate for all test conditions used in this investigation. Although the heating rate sensitivity of this steel is very low for stresses resulting in plastic deformation and failure in the 600° to 1000°F temperature range, no negative heating rate dependence was evident. An inversion in the loading rate dependence of this steel occurs between 400°F and 600°F with strength decreasing as loading rate increases. Relatively low negative values of loading rate or strain rate sensitivity resulted in inverse loading rate dependence throughout the 600° to 1000°F temperature range.

The inverse loading rate or strain rate dependence and the low heating rate dependence of this steel in the 600° to 1000°F temperature range are attributed to dynamic strain aging. The shorter times

available for diffusion of interstitial atoms to dislocations as heating or loading rates increase reduces the effective pinning of these dislocations and thus reduces strain-hardening which results in a tendency toward decreasing strength with increasing heating or loading rates. These dynamic aging effects are related to the amount and mobility of impurity atoms available in the martensite matrix, as well as the temperature and loading or heating rate. Dynamic strain aging is associated with the martensite matrix and does not involve any instability of the intermetallic precipitates formed during aging of the steel.

Yield strengths obtained in the heating rate tests are considerably lower than those obtained in the short-time elevated temperature tensile tests at temperatures above 600°F. This behavior is attributed to the difference in loading conditions in the two test procedures with respect to dynamic strain aging effects at temperatures below approximately 1000°F and to creep occurring in the heating rate tests at higher temperatures. The return of positive heating rate and loading rate dependence at temperatures above 1000°F results from increased rate sensitivity associated with recovery effects related to overaging phenomena with reversion of martensite to austenite also involved in the loading rate sensitivity in the tensile tests.

Aging treatments required to obtain a coexisting metastable precipitate and stable precipitate (850°F for 3 hours) and to obtain a stable precipitate (900°F for 3 hours), as determined from maraging kinetics and precipitate reversion studies in this investigation, did not significantly affect the heating rate or loading rate dependence of this 18Ni (30C) maraging steel. The 900°F aging treatment did result in slight improvement in yield strength in the 600° to 900°F temperature range in the tensile tests. These aging treatments had even less influence on the yield strength in the heating rate tests with the yield stress-temperature curves being essentially identical for the two aging treatments. The 900°F aging treatment did result in a slight increase in resistance to creep in the heating rate tests in the 900° to 1100°F temperature range.

The load-carrying ability of this steel is generally greater in the tensile test procedure than in the heating rate test procedure based on equal time under load in the 400° to 1000°F temperature range. Load-carrying ability for the heating rate test procedure decreases with elapsed time at all temperatures between 400°F and 1000°F, while load-carrying ability for the tensile test procedure decreases with time at 400°F, but increases with time at temperatures of 600° to 1000°F. It is apparent that it is impossible to predict load-carrying ability of this steel for a specified sequence of heating and loading from a different sequence of heating and loading even though times and temperatures under heating and loading are the same.

It is apparent that the utilization of strength data obtained in elevated temperature tensile tests on 18Ni (300) maraging steel at temperatures above 600°F could result in nonconservative design criteria for a solid propellant missile motor case or other structure in which loading and heating parameters correspond to those used in the heating rate test procedure. The influence of loading rate or strain rate on the mechanical behavior of this steel in conventional or short-time elevated temperature mechanical tests must also be considered for material selection or mechanical design applications since changes in the strain rate dependence of strength could be significant in many elevated temperature structural applications.

REFERENCES

1. Austin, C. W., Jr., Mechanical Behavior of 18Ni-9Co-5Mo (300 KSI) Maraging Steel Under Biaxial Stress and Rapid Heating, US Army Missile Command, Redstone Arsenal, Alabama, April 1965, Report No. RR-TR-65-3.
2. Austin, C. W., Jr., Mechanical Behavior of 18Ni-7Co-5Mo (250 KSI) Maraging Steel Under Biaxial Stress and Rapid Heating, US Army Missile Command, Redstone Arsenal, Alabama, August 1965, Report No. RR-TR-65-12.
3. Hauser, D. and Wright, J. W., Jr., "Tensile Behavior of High-Strength Alloys During Rapid Heating," Space Shuttle Materials, Vol. 3, Society of Aerospace Material and Process Engineers, National SAMPE Technical Conference, 1971, pp. 287-295.
4. Kendall, D. P., The Effect of Strain Rate and Temperature on Yielding in Steels, Watervliet Arsenal, Watervliet, New York, November 1970, Technical Report WAT-7061.
5. Brisbane, A. W., Hawn, J. M., and Ault, R. T., "Fracture Toughness and Delayed Failure Behavior of 18 Percent Nickel Maraging Steel," Mater Res and Stds., Vol. 5, 1965, pp. 295-405.
6. Shapiro, L., "Steels for Deep Quest and DSRV-1," Metal Progress, Vol. 93, No. 3, 1968, pp. 74-77.
7. Carter, C. S., "The Effect of Heat Treatment on the Fracture Toughness and Subcritical Crack Growth Characteristics of a 350-Grade Maraging Steel," Met. Trans., Vol. 1, 1970, pp. 1551-1559.
8. Peters, D. T., "Precipitate Reversion in 18Ni-Co-Mo Steels," Trans. TMS-AIME, Vol. 239, 1967, pp. 1981-1988.
9. Davenport, E. S. and Bain, E. C., "The Aging of Steel," Trans ASM, Vol. 23, 1935, pp. 1047-1096.
10. Peters, D. T. and Cupp, C. R., "The Kinetics of Aging Reactions in 18Ni Maraging Steels," Trans TMS-AIME, Vol. 236, 1966, pp. 1420-1429.
11. Miner, R. E., Jackson, J. K., and Gibbons, D. F., "Internal Friction in 18Ni Maraging Steels," Trans TMS-AIME, Vol. 236, 1966, pp. 1565-1570.
12. Cottrell, A. H., Dislocation Dynamics and Plastic Flow in Crystals, Oxford University Press, London, 1953.

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13. Leslie, W. C. and Sober, R. J., "The Strength of Ferrite and of Martensite as Functions of Composition, Temperature and Strain Rate," Trans. ASM, Vol. 60, 1967, pp. 459-484.
14. Owen, W. S. and Roberts, M. J., "Dynamic Aging Effects in Iron-Nickel-Carbon Martensite," Dislocation Dynamics, A. R. Rosenfield (Editor), McGraw-Hill Book Company, New York, 1968, pp. 357-378.
15. Steigerwald, E. A. and Hanna, G. L., "Spontaneous Strain Aging in High-Strength Steels," Trans ASM, Vol. 56, 1963, pp. 656-676.
16. Christian, J. W., "The Strength of Martensite," Strengthening Methods in Crystals, A. Kelly and R. B. Nicholson (Editors), John Wiley and Sons, Inc., New York, 1971, pp. 261-329.
17. Gillis, P. P., "Inverse Strain-Rate Effects," J. Appl. Phys., Vol. 40, 1969, pp. 2378-2380.
18. Johnston W. G. and Gilman, J. J., "Dislocation Velocities, Dislocation Densities, and Plastic Flow in Lithium Fluoride Crystals," J. Appl. Phys., Vol. 30, 1959, pp. 129-144.
19. Morris, J. G., "The Deformational Instability of Aluminum Alloys," Mater. Sci. Eng., Vol. 3, 1968, pp. 220-229.
20. Harrington, R. H., Stress-Aging: A New Treatment for Alloys, Part 6: Effect on Standard Tensile Properties of 18Ni-Maraging Steel, Watervliet Arsenal, Watervliet, New York, November 1967, Technical Report WVT-6744.
21. Wright, J. W., Jr., The Effect of Stress Aging on the Yield Strength of 250 Grade 18 Percent Nickel Maraging Steel Sheet, US Army Missile Command, Redstone Arsenal, Alabama, November 1968, Report No. RK-TR-68-18.